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**Recovery of Metal Values from
Hudson Bay Mining and Smelting
Oxide Residue**

Subcontract to Sherritt Gordon Ltd. by
Hudson Bay Mining and Smelting Co., Limited
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Dr. Mike Chalkley, Principal, Extractive Metallurgy, was in charge of the test program and was also responsible for editing the final report. Dr. Mike Collins, Research Metallurgist, directed the testwork and prepared the final report.

Significant contributions to the execution of the testwork were made by those listed below.

Mr. Russ Kofluk	Principal Technologist
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Mr. Jim Jost provided equipment preparation and maintenance services throughout the program. Miss Joan Didricksen, Mrs. Debbie Brun, Miss Sandra Dexter and Mrs. Natalie Walker provided the many pages of typed text. The Sherritt Analytical Services Department provided timely support during continuous operations.

Mr. Bill Burdenie, Consulting Engineer, looked after general management duties through most of the project.

1.0 INTRODUCTION

Under the Canada/Manitoba Mineral Development Agreement, funds were made available to Hudson Bay Mining and Smelting Company Limited to develop an economically viable and environmentally acceptable process for recovering metal values from zinc plant oxide residues stockpiled at Flin Flon. It is estimated that the stockpile amounts to some 450 000 tonnes (dry basis) with contained metal values worth U.S. \$300 to \$400/tonne.

The oxide residue, which has accumulated over many years, is the insoluble residue from the leaching of zinc oxide fume in zinc spent electrolyte. In addition to zinc, lead, copper and precious metal values, the residue contains substantial amounts of impurities, such as arsenic, antimony, germanium and fluoride, and as such is not suitable for treatment in existing zinc circuits. An independent treatment circuit, which will permit the separate recovery of contained zinc, lead, copper and precious metals, and rejection of the impurity elements is, therefore, required.

On July 30, 1985 HBM&S issued a request for a proposal to carry out Phase I of a two phase study leading to the definition of a viable process flowsheet for treatment of the oxide residue. The scope of the Phase I study included mineralogical analysis of the oxide residue, the development of a detailed preliminary flowsheet, and laboratory scale testwork to confirm the feasibility of the selected flowsheet. Potential alternatives for each step within the overall flowsheet were to be evaluated so that an economically attractive alternative could be identified.

On September 4, 1985 Sherritt submitted Proposal 6637 to HBM&S to carry out Phase I for the project in two stages. Phase Ia would consist of mineralogical analysis and batch process testwork, together with a preliminary engineering and economic evaluation of alternative processing routes, while Phase Ib would consist of continuous miniplant testing of the selected flowsheet.

On March 12, 1986 HBM&S accepted Sherritt's proposal and authorized work to proceed on Phase Ia of the program, under DSS File No. 06SQ23440-5-9220, Contract No. 0SQ85-00312. The final report on Phase Ia was issued in September 1987. The test program actually carried out by Sherritt in consultation with HBM&S was larger in scope than had originally been proposed, and in consequence the preliminary engineering and economic evaluation was not carried out in Phase Ia.

The Phase Ia test program resulted in the identification of an effective leaching and flotation flowsheet for the recovery of copper to a sulphide concentrate, and lead to a relatively clean sulphate residue, with silver and gold distributed between the two flotation products. However, the preferred method for treating the leach solution, which in addition to zinc, contains high levels of iron and arsenic, had not been established. Selective

recovery of zinc by solvent extraction using D2EHPA appeared promising technically, but was uneconomic due to a high consumption of sodium hydroxide. The alternative approach of precipitating iron from the zinc bearing solution either as iron hydroxide, or as jarosite, merited further investigation.

On October 7, 1987 Sherritt submitted Proposal 6658 to HBM&S to carry out further batch testwork on these aspects of the provisional process flowsheet. The additional work scope was designated Phase Ib-1, while the original scope proposed for Phase Ib in the 1985 proposal was redesignated Phase Ib-2.

On November 26, 1987 HBM&S accepted Sherritt's proposal and authorized work to proceed on Phase Ib-1 of the program under Purchase Order No. 712883. The final report on Phase Ib-1 was issued in August, 1988.

The flowsheet tested in Phase Ib-1 of the test program is shown in Figure 1.1. In this flowsheet, the oxide residue is treated with sulphuric acid and elemental sulphur in an atmosphere of sulphur dioxide to dissolve zinc, iron, arsenic and other impurity elements. A two stage, countercurrent leach is preferred to produce a low acid discharge. The source of sulphuric acid for the leach may conveniently be return zinc spent electrolyte. The reducing leach residue is subjected to flotation for separation of copper to the concentrate, and lead to the tailing. The reducing leach solution proceeds to a two stage iron removal step for precipitation of iron and impurity elements to leave a clean zinc sulphate solution.

The Phase Ib-1 testwork demonstrated that zinc, lead and copper could be separated from the oxide residue according to the flowsheet given in Figure 1.1, and that iron and arsenic could be effectively removed from the zinc sulphate leach liquor, by either a jarosite or a goethite type of iron removal process. However, the metal contents of three product streams, zinc sulphate solution, lead sulphate residue, and copper-zinc sulphides, were not yet of significant market value due either to low metal grade or to high impurity levels.

On October 24, 1988 Sherritt submitted Proposal 6682 to HBM&S for Phase Ic of the study. Phase Ic would replace the Phase Ib-2 program defined in Proposal 6658 and would consist of batch and continuous tests to establish the viability of an expanded treatment process, and the preparation of cost estimates for a commercial plant.

The revised treatment flowsheet is shown in Figure 1.2. Hot acid leach, neutralization-reduction and zinc flotation steps would be added ahead of the reducing leach to give better separation of copper and zinc sulphides and to increase the concentration of zinc in the solution. Another flotation step would be added to separate an upgraded lead concentrate from the copper flotation tailing.

On February 28, 1989 HBM&S accepted Sherritt's proposal and authorized work to proceed under HBM&S Purchase Order 714736. Funding for the study was provided to HBM&S by CANMET under Contract Serial Number 0SQ85-00312.

Initial batch tests were carried out using feed materials from earlier phases of the project. New samples of oxide residue, zinc concentrate, zinc hydroxide sludge and purified neutral leach solution were obtained from HBM&S in March and May and batch testwork continued through June. The continuous testwork was conducted in July and August, and additional batch tests continued through December, 1989.

This report provides an overall compilation and interpretation of the process data generated during the Phase 1c batch and continuous test programs.

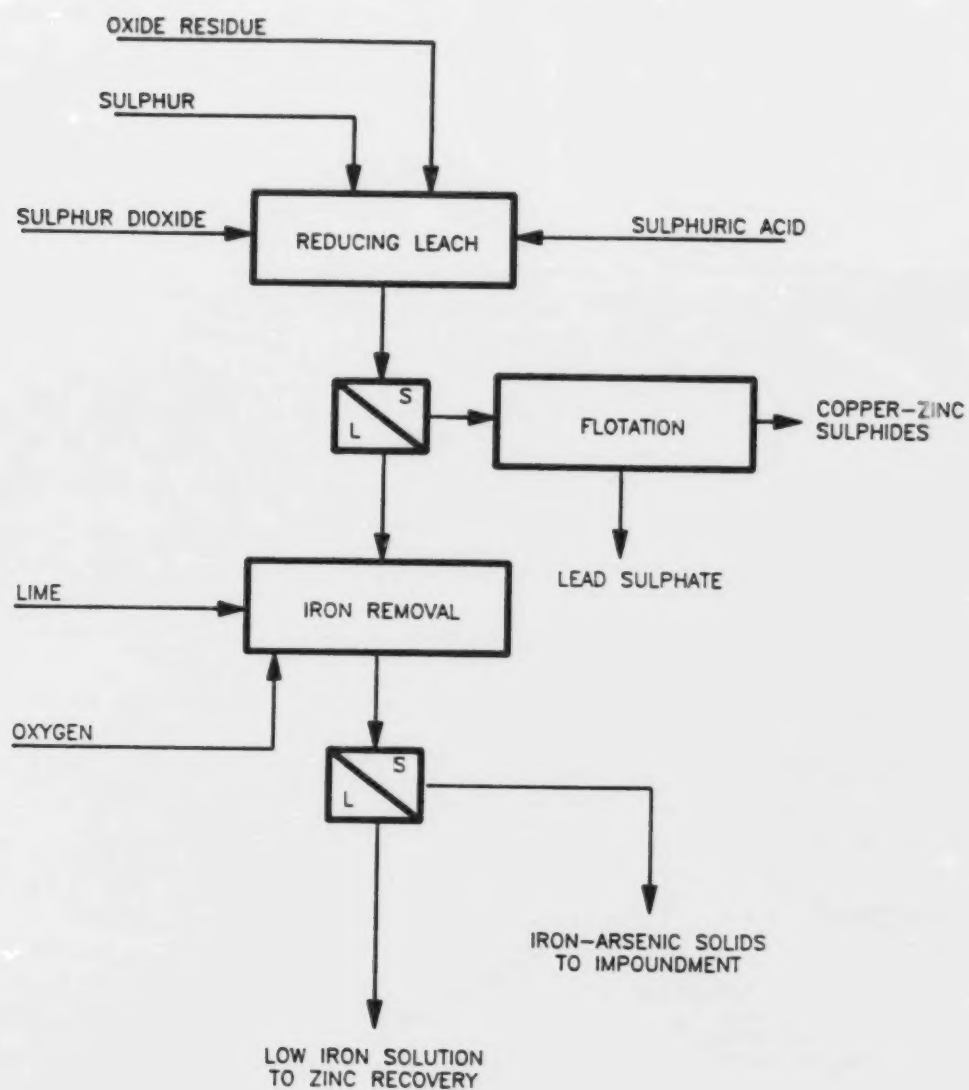


Figure 1.1 Phase Ib-1 Oxide Residue Treatment Flowsheet

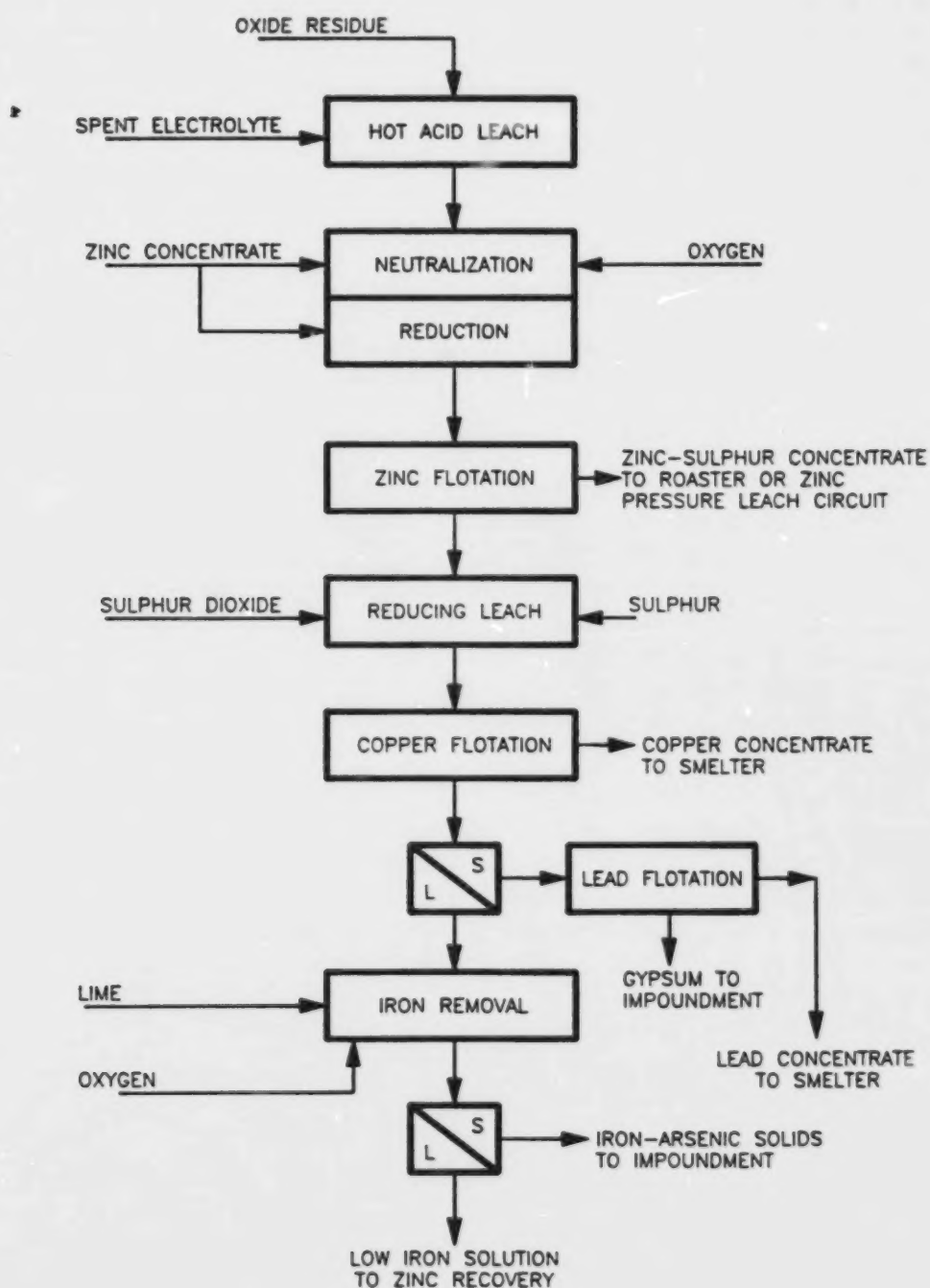


Figure 1.2 Phase Ic Oxide Residue Treatment Flowsheet

2.0 SUMMARY

This report describes the results of a program of batch and continuous miniplant testwork to demonstrate a process for separation of zinc, lead, copper and precious metal values from HBM&S stockpiled zinc oxide fume leach residue.

In February, 1989 HBM&S engaged Sherritt Gordon Limited to proceed with Phase Ic of the process development studies. The major objective of this phase of the test program was to demonstrate a new flowsheet for the oxide residue treatment process that would allow for the recovery of separate, high grade concentrates of zinc, lead and copper, and a high strength zinc sulphate solution.

The proposed oxide residue treatment process is described in detail in Section 3. In the process, the stockpiled oxide residue is contacted with return zinc spent electrolyte at atmospheric pressure to partially dissolve zinc, copper, iron and impurity elements. It is of particular importance to the recovery of copper that sulphide copper in the residue is leached in this hot acid leach step. The resulting slurry is treated with zinc concentrate under oxygen sparging conditions at atmospheric pressure to consume the excess acid from the previous step and to increase the concentration of zinc in the solution. Additional concentrate is fed to the slurry in the absence of oxygen to reduce ferric iron in solution to the ferrous state. Excess zinc concentrate, zinc sulphide in the original feed and elemental sulphur produced in the reaction of zinc concentrate with acid and ferric iron in the neutralization-reduction step are recovered by flotation. This zinc flotation concentrate is suitable as feed to a zinc pressure leach circuit or a roaster.

The zinc flotation tailing slurry is treated with sulphur dioxide and elemental sulphur at a pressure of about 200 kPa to further extract zinc, copper and iron, to reprecipitate copper as copper sulphide, and to convert lead in jarosite to lead sulphate.

The slurry resulting from the reducing leach with sulphur dioxide is subjected to a second flotation step to separate a copper sulphide concentrate. This flotation concentrate is suitable as feed to a copper smelter.

In a third flotation step, the copper flotation tailing solids are treated for recovery of a lead sulphate flotation concentrate, which is suitable as feed to a lead smelter. The lead flotation tailing, comprised of gypsum and other gangue minerals, is stockpiled.

The process solution, containing most of the zinc in the head material, is treated with lime or zinc hydroxide sludge in a two stage process for precipitation of iron and impurity elements from solution. The solution is maintained at about pH 4 or less in the first stage to limit the coprecipitation of zinc. After liquid-solid separation, the iron cake is impounded and the solution proceeds to a second stage of iron removal at pH 5. The resulting slurry is clarified and the solids recycled to the first stage of the iron removal step.

for recovery of precipitated zinc. The low iron zinc sulphate solution may be treated in the existing zinc plant.

Precious metal values in the oxide residue head material are distributed between the three flotation concentrates in the process, where they may be recovered along with the contained zinc, copper, or lead values.

The test program consisted of preliminary batch tests followed by continuous hot acid leach, neutralization-reduction, zinc flotation, reducing leach and copper flotation tests. The continuous hot acid leach and neutralization-reduction tests were carried out concurrently and the other tests separately. Batch lead flotation and iron removal tests were carried out after completion of the continuous testwork.

2.1 FEED MATERIALS

Two samples of stockpiled oxide residue were treated in the Phase Ic testwork. The chemical analysis for the sample used for the continuous testwork is summarized below.

Sample	Analysis, %							g/t		
	As	Cd	Ca	Cu	Fe	Pb	Zn	F	Ge	Ag
Oxide Residue	1.3	0.1	6.4	1.6	14.5	13.7	11.5	121	88	163

The principal minerals identified in the oxide residue are anglesite, jarosite and zinc ferrite. Other contained minerals include gypsum, sphalerite and covellite.

2.2 HOT ACID LEACH

The continuous hot acid leach circuit was operated for 55 h. A total of 195 kg of oxide residue was processed through the circuit, which consisted of a train of four heated, agitated tanks arranged in cascade. The retention time and the acid addition rate were both varied during the test and the temperature was maintained at 90° C. The results for the two operating periods are summarized below.

Period	Ret. Time h	H ₂ SO ₄ Addition kg/t	Solution Analysis, g/L				Extraction, %		
			Cu	Fe	H ₂ SO ₄	Zn	Cu	Fe	Zn
1	6.5	377	3.2	13.3	53.4	60.3	65	32	25
2	12.7	478	3.2	18.9	48.4	68.6	69	46	34

Under the preferred conditions in Period 2, the acid consumption was about 300 kg H₂SO₄ per tonne of oxide residue and the extractions were about 70% Cu, 45% Fe and

35% Zn. Although the total retention time was 12 hours during Period 2, reaction was essentially complete by the third tank in the train, representing a 9 h retention time.

All of the acid consumed in the continuous hot acid leach test was provided by simulated zinc spent electrolyte. A terminal acidity of at least 50 g/L H_2SO_4 was desirable to promote rapid leaching and to prevent precipitation of iron from the solution.

2.3 NEUTRALIZATION-REDUCTION

The continuous neutralization-reduction circuit was operated for 47.5 h. 603 L of hot acid leach slurry and 77 kg of zinc concentrate were fed to the circuit. Oxygen was sparged into the first four tanks of the circuit, which consisted of a train of five heated and agitated tanks arranged in cascade. No oxygen was added to the last tank, to enable reduction of ferric iron in solution with excess zinc concentrate.

Acid consumption was low in the early stages of the test, but a satisfactory solution composition was obtained after suitable adjustments to the reagent addition rates, the agitation, and the retention time. Average results for the last two periods of operation are summarized below.

Period	Retention Time h	Concentrate Addition kg/t	Solution Analysis, g/L				
			Cu	Fe^{2+}	Fe	H_2SO_4	Zn
3 and 4	6.4	520	3.43	14.9	21.4	18.4	84.6

A retention time of 6 h was sufficient at 90°C to decrease the acid concentration in the solution from about 50 g/L H_2SO_4 to between 15 and 20 g/L H_2SO_4 . The concentration of ferric iron in the discharge solution averaged 6.5 g/L during Periods 3 and 4, but was readily reduced to between 2 and 5 g/L in the earlier operating periods. Extraction of zinc from the concentrate was between 60 and 70% in Periods 3 and 4 and zinc pickup to the solution averaged 15 g/L.

Copper extracted into the solution during the hot acid leach step was not precipitated during neutralization-reduction. Control of the operating conditions to prevent copper precipitation did not prove difficult during the continuous test, and a small net extraction of copper, originating from the added zinc concentrate, was observed.

The zinc concentrate addition rate was not optimized during the continuous test, but it would appear that at least 400 kg of concentrate is required per tonne of stockpiled oxide residue to meet the process objectives.

2.4 ZINC FLOTATION

The zinc flotation tests demonstrated that excess zinc concentrate added during neutralization-reduction and sphalerite contained in the oxide residue could be recovered by flotation. No flotation reagents were required since the residual zinc sulphide was intimately mixed with elemental sulphur produced by partial leaching of zinc sulphide in the previous steps, which assisted flotation. Results for the continuous flotation test are summarized below.

Fraction	Analysis, %						
	Cu	Fe	Pb	S ^o	S(SO ₄)	S	Zn
Feed	0.82	10.4	10.6	5.4	6.0	22.3	24.2
Cleaner Concentrate	1.06	9.6	2.8	9.1	0.7	37.2	43.8
Rougher Concentrate	1.02	10.1	6.4	5.1	2.1	31.0	39.1
Scavenger Concentrate	0.96	10.0	10.1	2.9	3.7	25.8	33.1
Scavenger Tailing	0.59	10.8	17.3	3.4	9.9	11.2	7.3

Fraction	Weight kg	Distribution, %					
		Cu	Fe	Pb	S ^o	S	Zn
Cleaner Concentrate	64.6	55.8	38.4	10.2	65.3	70.0	80.8
Scavenger Tailing	92.0	44.2	61.6	89.8	34.7	30.0	19.2

The scavenger operation was not very efficient in the continuous test, and the scavenger tailing solids contained 7.3 % Zn on average compared with about 6% Zn in batch tests. The residual 1.3 % Zn as zinc sulphide contained in the continuous flotation tailing was recovered along with copper sulphide in the continuous copper flotation test, and was a significant diluent in this concentrate. Careful attention must be paid to the zinc scavenger operation to allow for production of a high grade copper concentrate.

The continuous cleaner concentrate contained 44% Zn and 9% elemental sulphur. With optimization of the zinc concentrate addition rate in the neutralization-reduction step, the zinc flotation concentrate will have a higher concentration of elemental sulphur and a correspondingly lower concentration of zinc. This flotation concentrate is suitable for feed to a zinc pressure leach circuit for recovery of the contained zinc and sulphur values.

A concentrate containing 41% Zn by weight was obtained by flotation of hot acid leach slurry directly. This rougher-scavenger concentrate contained 26% of the zinc in the hot acid leach residue. It is clear from this batch test that a significant proportion of the zinc in the oxide residue feed is present as sphalerite, and much of this sphalerite survives the hot acid leach.

2.5 REDUCING LEACH

Zinc flotation tailing slurry was treated in a six compartment horizontal autoclave, at 100°C, under 100 kPa sulphur dioxide overpressure in the continuous reducing leach circuit. Elemental sulphur was added to the feed slurry to assist in the precipitation of copper. A total of 304 L of zinc flotation tailing slurry was processed through the circuit, which was operated for 44 h. Results for the test are summarized below.

Period	Ret. Time h	Discharge Method	Reagents kg/t		Solution Analysis, g/L				Extraction %	
			S	SO ₂	Cu	Fe	H ₂ SO ₄	Zn	Fe	Zn
1	4.3	Precooled	13.4	169	0.01	41.6	13.6	84.5	93.0	85.7
2	4.3	Precooled	9.0	158	0.01	43.5	11.7	95.1	92.2	85.3
3	4.3	Flashed	4.5	115	0.02	45.6	5.9	93.1	90.2	84.7
4	4.4	Flashed	9.0	117	0.01	40.8	5.3	88.6	91.8	85.1

The retention time in the autoclave was about 4 h. The elemental sulphur addition rate varied between 4.5 and 13.4 kg per tonne of oxide residue. Copper precipitation was independent of the sulphur addition rate, and was essentially quantitative throughout the test. Subsequent examination of the feed solids revealed that sulphur remaining from the previous steps was sufficient to precipitate all of the copper in solution in the reducing leach. With more efficient recovery of zinc and sulphur in zinc flotation, it is expected that the sulphur requirement in the reducing leach will be between 5 and 10 kg/t.

The discharge slurry was switched from indirect cooling in a coil to flash cooling after the second period of operation. The concentration of acid in the discharge solution decreased by about 8 g/L H₂SO₄ as a result of this change, corresponding with evolution of sulphur dioxide from the flashed slurry. Sulphur dioxide consumption decreased in the second period because the discharge vapour was recycled. The lower acid content of the flashed slurry also results in lower reagent requirements in the iron removal step.

The reducing leach discharge solution contained about 43 g/L Fe and about 91 g/L Zn. Virtually all of the iron was in the ferrous state, which is suitable for treatment of the solution in the iron removal step.

Net extractions of iron and zinc from the stockpiled oxide residue averaged 92% and 85%, respectively, during the continuous test, compared with 93 % and 91% in batch tests. The lower zinc extraction in the continuous test was related to residual zinc sulphide in the zinc flotation tailing feed to the continuous reducing leach. Treatment of the leach residue in the copper flotation step yielded a flotation tailing containing 1.3% Zn, representing an overall zinc extraction of 94%.

2.6 COPPER FLOTATION

Copper sulphide precipitated in the reducing leach was recovered by flotation in the copper flotation step. Eleven batch tests were carried out followed by the continuous test which treated 262 L of slurry containing 51 kg of solids.

Copper recovery in the batch tests with the continuous reducing leach discharge as the feed was typically 95% with Z200 added to assist the flotation. The rougher-scavenger concentrates contained 20 to 25% by weight copper. Analyses for the cleaner concentrate recovered in Batch Test 10 are given below.

Fraction	Analysis, %				
	Cu	Pb	SiO ₂	S	Zn
Cleaner Concentrate	26.9	7.1	4.4	35.7	12.8

The low grade of the copper concentrate was a result of aging of the slurry, which tended to result in poor rejection of lead sulphate and gangue minerals, and the presence of a significant quantity of zinc sulphide in the feed as a result of inefficient scavenging of zinc sulphide in the earlier zinc flotation step.

Results for the continuous copper flotation test were similar to the batch tests, although scavenging of copper was less efficient in the continuous test. The results are summarized below.

Fraction	Analysis, %					
	Cu	Fe	Pb	S(SO ₄)	S	Zn
Feed	2.8	1.2	22.1	11.9	15.1	2.7
Cleaner Concentrate	16.0	4.2	7.4	4.3	30.5	13.3
Rougher Concentrate	7.3	2.5	19.4	8.4	18.7	7.8
Scavenger Concentrate	2.6	1.2	23.3	11.2	13.7	3.3
Scavenger Tailing	0.4	0.8	25.5	12.3	12.8	1.3

Fraction	Distribution, %					
	Cu	Fe	Pb	S(SO ₄)	S	Zn
Cleaner Concentrate	87.0	47.2	4.9	5.9	29.9	64.5
Scavenger Tailing	13.0	52.8	95.1	94.1	70.1	35.5

Additional batch tests were carried out after completion of the continuous testwork to establish that a high grade copper concentrate could be obtained. Slurry from the continuous neutralization-reduction step was treated in batch zinc flotation and reducing

leach steps, and copper flotation was carried out immediately after completion of the reducing leach test. Results of the batch copper flotation tests are summarized below.

Fraction	Analysis, %			Distribution, %		
	Cu	Pb	Zn	Cu	Pb	Zn
Cleaner Concentrate	49.0	0.2	1.1	87.9	0.1	3.8
Rougher-Scavenger Concentrate	36.4	11.1	3.5	0.7	3.7	8.8
Rougher-Scavenger Tailing	0.3	18.3	1.5	11.4	96.2	87.3

Improved flotation of zinc in the batch zinc flotation tests allowed for good separation of copper and zinc to their respective flotation concentrates. Flotation of the reducing leach slurry immediately after discharge from the autoclave allowed for good rejection of lead and gangue minerals. The copper flotation concentrate contained 49% Cu, 1.1% Zn and 0.2% Pb. Copper recovery was 88%.

2.7 LEAD FLOTATION

The continuous copper flotation tailing slurry was thickened and the thickener underflow providing the feed for lead flotation tests. In the first series of tests, the copper flotation tailing solids were washed and repulped with water, and the pH of the solution was increased by addition of sodium hydroxide or lime. Sodium sulphide was then added to sulphidize the surface of the lead sulphate particles, which were floated from the gangue with the assistance of a xanthate as the collector. Results for these tests are summarized below.

A high grade lead concentrate was obtained at pH 8.6, but good recovery of lead to the concentrate was not achieved until the pH was increased to between 8.9 and 9.8. Calcium was successfully rejected to the tailing in all of the tests. The scavenger tailing typically contained 20% by weight calcium, or about 85% gypsum.

Test	pH	Analysis, %				Distribution, %			
		First Concentrate		Scavenger Tailing		Combined Concentrate		Scavenger Tailing	
		Ca	Pb	Ca	Pb	Ca	Pb	Ca	Pb
1	8.6	0.3	53	16	13	1	66	99	34
2	8.9	2.7	45	20	3	13	95	87	5
3	9.7	0.4	58	20	2	11	94	89	6
4	9.8	0.4	53	23	2	7	95	93	5

Calcium hydrosulphide was used in place of sodium sulphide in six batch tests. Although concentrates containing up to 60% Pb were obtained, the lead recovery was typically less than 90%, compared with about 95% in tests with sodium sulphide as the sulphidizing agent.

Lead was also recovered by flotation with hexadecyl ammonium chloride as the collector. The batch tests were carried out with 5 g/L H_2SO_4 in the solution, which is the acid concentration of the copper flotation tailing solution. A cleaner concentrate containing 68% Pb by weight was obtained, but lead recovery was only 84%. Use of additional reagent resulted in flotation of a large portion of gypsum along with the residual lead. Additional tests with hexadecyl ammonium chloride and other collectors are required to establish if direct flotation of the copper flotation tailing slurry is a viable process option. Direct treatment of this slurry would save a liquid-solid separation and washing step.

2.8 IRON REMOVAL

Copper flotation tailing thickener overflow solution was the feed for batch iron removal tests. The tests were carried out in two stages. Zinc hydroxide sludge was added in the first stage to increase the solution pH to about 3.5 and after liquid-solid separation, lime was added to the solution to increase the pH to 5.0 in the second stage. Oxygen was sparged into the slurry in both stages to oxidize ferrous iron. Results for the two batch tests which were carried out at atmospheric pressure and 85°C are summarized below.

Test	Analysis						
	Iron Cake, %				Second Stage Solution, g/L		
	Ca	Fe	S(SO ₄)	Zn	As	Fe	Zn
1	14.2	20.9	12.5	0.8	0.0009	<0.0005	85.1
2	15.3	22.5	13.5	1.1	0.0003	<0.0005	114

The solids from the first stage iron removal contained about 20% Fe and 1% Zn. The zinc loss to the cake was about 5% of the zinc in the oxide residue feed to the process. The product solution contained less than 1 mg/L Fe, which is a suitable feed to conventional zinc purification and electrowinning steps.

2.9 BEHAVIOUR OF IMPURITIES

Arsenic, fluoride and germanium largely reported to the iron removal cake, leaving 6 mg/L F and less than 1 mg/L As and Ge in the iron free zinc sulphate solution. Chloride was not precipitated in iron removal, but the chloride content of the feed materials is low, and the iron free zinc sulphate solution contained only 17 mg/L Cl.

2.10 CONCLUSIONS AND RECOMMENDATIONS

A process for treatment of the HBM&S stockpiled oxide residue has been investigated in a series of batch and continuous tests. The laboratory studies have demonstrated

that zinc, lead, copper and precious metals may be separated from the oxide residue in a form or forms amenable to the recovery of these metal values.

Zinc is recovered as a zinc sulphate solution suitable as feed to conventional purification and electrowinning steps, and as zinc sulphide in a flotation concentrate suitable as feed to a zinc pressure leach plant. Total recovery of zinc is about 85%.

Copper is recovered in about 90% yield to a high grade concentrate suitable as feed to a copper smelter.

Lead is recovered in about 95% yield to a high grade concentrate suitable as feed to a lead smelter.

Silver recovery is about 95%, with the silver values split between the zinc, lead and copper concentrates.

A large quantity of zinc concentrate is required for acid neutralization purposes, and to increase the concentration of zinc in the process solution. If the concentrate contains copper, it would be recoverable in the copper concentrate produced in the process.

The major reagent requirements per tonne of oxide residue feed are about 100 kg/t sulphur dioxide, 10 kg/t elemental sulphur and 100 kg/t lime. The bulk of the lime requirement can be provided by zinc hydroxide sludge, which would also serve to increase the concentration of zinc in the product zinc sulphate solution.

Sherritt is confident that the testwork carried out in this phase of the oxide residue project has demonstrated the technical viability of the current treatment flowsheet. Some additional batch tests are required, especially for the lead flotation step, before the integrated flowsheet should be tested on a continuous basis.

3.0 PROCESS SELECTION

Based on the results of the Phase Ic process development program and preliminary process engineering, as well as considerable Sherritt experience, a process is presented for recovery of metal values from HBM&S stockpiled oxide residue. The process has been developed assuming integration with the proposed zinc pressure leach plant at Flin Flon. The process will treat stockpiled oxide residue to realize a high recovery of zinc, copper, and lead as well as conversion of iron and arsenic to a disposable residue.

The process consists of the following steps:

oxide residue reclaim;

hot acid leach;

neutralization-reduction;

zinc flotation;

reducing leach;

copper flotation;

lead flotation; and

iron removal.

Stockpiled oxide residue is reclaimed from HBM&S's existing tailings pond, repulped with recycle liquor and transferred to a feed/surge tank with a capacity to hold 18 hours of feed slurry.

The repulped residue is leached under highly acidic conditions in an atmospheric hot acid leach utilizing hot zinc spent electrolyte as the lixiviant. The hot acid leach consists of four brick lined tanks in series. Slurry overflows from the last tank into another train of five tanks which make up the neutralization-reduction step.

In the neutralization-reduction step, the hot acid leach discharge slurry is contacted with zinc concentrate and oxygen. Sulphuric acid is neutralized and ferric iron is reduced to the ferrous state. The resulting slurry, which contains about 15 g/L H_2SO_4 in solution, is advanced to the zinc flotation step.

The zinc flotation circuit consists of rougher, scavenger, and cleaner cells. Zinc flotation concentrate is filtered on a belt filter, and the cake is transferred to the zinc

pressure leach plant. The flotation tailing slurry is transferred to a surge tank before advancing to the reducing leach circuit.

The reducing leach consists of a single six compartment autoclave and a flash tank. Elemental sulphur and Lignosol are blended into the feed slurry which is pumped through a preheater into the first compartment of the autoclave. Leaching is conducted at a temperature of 100°C and a pressure of 100 kPa (gauge) utilizing sulphur dioxide. The autoclave discharge slurry is flashed to release excess sulphur dioxide. The flashed slurry drains into a surge tank before it advances to the copper flotation step.

The copper flotation circuit consists of rougher, scavenger, and cleaner cells. The copper flotation tailing slurry is transferred to a thickener which separates the bulk of the liquor from the residue which advances to the lead flotation circuit. The thickener overflow is advanced to the iron removal circuit. The flotation concentrate is filtered and washed to remove entrained zinc liquor and transferred to the existing copper flotation plant for blending with the main feed to the copper smelter.

The lead flotation circuit also consists of rougher, scavenger, and cleaner cells. The copper flotation tailing thickener underflow is filtered and washed prior to treatment in the rougher cells. The filter cake is repulped with water and the resulting slurry is neutralized with lime and the lead sulphate particles are activated with calcium hydrosulphide. Activated lead sulphate particles are consequently floated to produce a high grade lead cake which is filtered, washed, and dried for sale to a lead smelter. The lead flotation tailing, comprised principally of gypsum, is combined with gypsum residues produced by other operations at the HBM&S refinery.

Overflow from the copper flotation tailing thickener is fed into a two stage iron removal circuit. The first stage utilizes zinc sludge produced in the waste water treatment circuit for solution neutralization to about pH 3.5. Lime is the reagent used in the second stage where the iron precipitation is completed at pH 5. By conducting the iron removal in two stages, the loss of zinc to the discard iron cake is minimized. The circuit consists of three tanks in cascade followed by a thickener and two additional tanks in cascade, followed by a second thickener. The solution from the second thickener is pumped to the zinc purification circuit. Solids in the first thickener underflow are pumped to a filter circuit which removes entrained zinc liquor from the iron residue before it is discarded to a tailings pond.

A description of a commercial facility based on the process outlined above is presented in this section. Equipment sizes suggested in this chapter are not definitive for the purposes of design of a commercial facility, but are provided for orientation only. The overall process is presented schematically opposite each page of text in this section for the convenience of the reader.

3.1 OVERALL BASIS

3.1.1 Plant Capacity

The treatment facilities are designed to treat approximately 130 t/d of oxide residue. An annual capacity of 40 000 tonnes of oxide residue provides for 10 years of operation before the existing stockpile is exhausted.

3.1.2 Chemical Analysis of the Feed Material

Since the oxide residue is the tailing resulting from operation of the zinc oxide fume leach circuit by HBM&S over many years, the composition varies throughout the stockpile. The expected range and average analyses of the important components are tabulated below.

Component	Weight % Dry Basis (*g/t)		
	Range		Average
Arsenic	1.3	to 5.5	3.0
Cadmium	0.12	to 0.19	0.15
Calcium	6.2	to 6.4	6.3
Copper	1.4	to 2.2	1.5
Iron	10.9	to 14.5	12.0
Lead	13.7	to 16.6	15.0
Silica	3.3	to 4.7	4.0
Sulphate	22.5	to 26.1	25.0
Zinc	8.9	to 14.1	11.5
Germanium*	54	to 100	75
Gold*	2.1	to 2.4	2.3
Silver*	105	to 163	125

3.1.3 Process Objectives

To achieve a viable process for the treatment of the stockpiled residue, a flowsheet with the following objectives has been pursued:

a high recovery of zinc to either a zinc sulphide concentrate or an iron free zinc sulphate solution with a minimum concentration of 100 g/L zinc;

a high recovery of copper to a separate copper concentrate; and

a high recovery of lead to a separate lead concentrate grading at least 40% lead.

These objectives are predicated on the basis of the dollar value of contained metals in the oxide residue. Zinc, lead, and copper comprise between 80% and 90% of the valuable elements, at current prices.

3.2 OXIDE RESIDUE RECLAIM

The objective of the oxide residue reclaim step is to remove the residue from the tailings pond and convert it into a slurry of 45% by weight solids. The capital cost estimate includes an allowance for residue reclaim, including a 6 m x 6 m storage tank for 18 h of feed.

3.2.1 Process Control Highlights

Recycle of liquor from the oxide residue treatment circuit is desirable for repulping the feed solids in the reclaim section. Dilution with process water should be avoided to maximize the concentration of zinc in the solution.

3.3 HOT ACID LEACH

The objectives of the hot acid leach are:

to decompose the zinc ferrite, generating a concentrated solution of zinc and ferric sulphates;

to oxidize copper and zinc sulphides, utilizing the ferric iron produced from zinc ferrite decomposition as the oxidizing agent; and

to minimize the formation of plumbojarosite by maintaining a high solution acid concentration.

3.3.1 Process Description

A rubber lined pump transfers the oxide residue slurry into the first of a series of four pyroflex/brick lined overflow tanks. Each tank is about 4 m diameter and 4.5 m high to provide a total of nine hours retention time. Axial flow agitators are fitted to each tank for the purpose of solids suspension and moderate intensity mixing between the acidic lixiviant and the oxide residue. A heat exchanger preheats the lixiviant (zinc spent electrolyte) such that at least 85°C is maintained in the hot acid leach tanks. The preheated electrolyte is added to the first reaction tank and provides the source of sulphuric acid consumed by the reactions. A terminal acidity of at least 50 g/L H₂SO₄ prevents the reprecipitation of iron in the form of plumbojarosite.

After about nine hours of leaching, the hot acid leach slurry overflows into the first tank of the neutralization-reduction section.

3.3.2 Operating Conditions and Expected Results

Operating Conditions

Pressure	Atmospheric
Temperature	85°C
Retention Time (nominal)	9 hours
Feed Slurry Solids Content	45% by weight
Discharge Slurry Solids Content	15% by weight

Spent Electrolyte

H ₂ SO ₄	135 g/L
Zinc	60 g/L

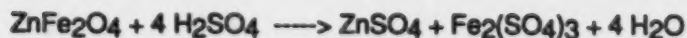
Hot Acid Leach Discharge Solution

Iron (total)	14 g/L
Zinc	64 g/L
H ₂ SO ₄	50 g/L
Copper	3 g/L
Arsenic	5 g/L

Extractions and Conversions

Zinc Extraction	35%
Copper Extraction	70%
Iron Extraction	45%

Reactions



3.3.3 Process Control Highlights

The treatment of oxide residue in the hot acid leach consists of mixing acidic solution (zinc spent electrolyte) and the residue for a period of nine hours at a temperature of 85°C.

Control of oxide residue mass flow and electrolyte volumetric flow is required to ensure that sufficient acid is available to decompose zinc ferrite and to achieve the target acid concentration in hot acid leach discharge slurry. A high acid concentration during hot acid leaching favours the zinc ferrite reaction and is important for the prevention of iron reprecipitation in the form of plumbojarosite.

The target hot acid leaching temperature is achieved by preheating the cellhouse return electrolyte. The leach tanks would be insulated to reduce heat losses.

3.4 NEUTRALIZATION-REDUCTION

The objectives of the neutralization-reduction step are:

- to neutralize the sulphuric acid in the hot acid leach solution so as to minimize the reagent requirements in the iron removal step;

- to reduce the ferric iron in solution to ferrous in order to minimize the generation of sulphuric acid in the reducing leach step;

- to increase the zinc concentration in solution by using zinc concentrate as the reagent; and

- to prevent dissolved copper from precipitating and contaminating the zinc concentrate recovered in the following flotation step.

3.4.1 Process Description

Discharge slurry from the hot acid leach step overflows directly into the first tank of a series of five pyroflex/brick lined tanks. Each tank is 3.2 m diameter and 4.0 m high to provide a total of six hours retention time. Agitation is provided by an axial over radial impeller configuration in the first four tanks, and by dual axial impellers in the fifth tank. Oxygen is sparged beneath each of the agitators within the first four tanks. The agitation must be sufficient to provide intimate mixing between the zinc concentrate reagent, acidic lixiviant, and the injected oxygen gas. Zinc concentrate is pumped as a 70% solids slurry into the first, third and fifth tanks. Each tank is also provided with low pressure steam injection dip pipes to maintain a temperature of 90°C.

After about six hours of leaching, the neutralization-reduction slurry is pumped to the zinc flotation rougher cells.

3.4.2 Operating Conditions and Expected ResultsReagents and Conditions

Zinc Concentrate	400 kg/t of oxide residue
Oxygen Gas (99.5% Pure)	90 kg/t of oxide residue
Pressure	Atmospheric
Temperature	90°C
Retention Time (nominal)	6 hours

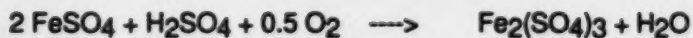
Solution Analyses

Neutralization-Reduction Discharge Solution

Iron (total)	18 g/L
Zinc	98 g/L
H ₂ SO ₄	15 g/L
Copper	3 g/L
Arsenic	5 g/L

Extractions and Conversions

H ₂ SO ₄ neutralized	70% of input
Iron reduced	75% of Fe ³⁺ input

Reactions3.4.3 Process Control Highlights

Process control is exercised to achieve four results.

These are:

to reduce the ferric iron concentration from 14 g/L in the feed slurry to about 3.5 g/L;

to reduce the acid concentration from 50 g/L in the feed slurry to about 15 g/L;

to increase the zinc tenor from 64 g/L in the feed slurry to about 100 g/L;

to prevent copper precipitation from solution.

Zinc concentrate and oxygen flow control and distribution provide the means to control the acid concentration and to increase the zinc tenor in solution. Control of zinc concentrate mass flow to the fifth tank provides the means to achieve the target ferric iron concentration. Sufficient ferric iron in solution (at least 2 g/L Fe^{3+}), ensures minimal precipitation of copper from the solution.

3.5 ZINC FLOTATION

The objective of the zinc flotation step is to separate the neutralization-reduction discharge solids into a zinc rich concentrate and a lead rich residue. The concentrate is filtered before transfer to the pressure leach concentrate regrind circuit.

3.5.1 Process Description

The neutralization-reduction residue is subjected to a rougher flotation followed by further upgrading of the rougher concentrate in a cleaner stage. Rougher tailing is treated in scavenger flotation stages to minimize the sulphide and sulphur content in the oxidic residue which reports to the scavenger tailing.

The neutralization-reduction discharge slurry, scavenger concentrate, and cleaner tailing are blended and then fed to a 3.0 m³ rougher flotation cell. The pulp density of the flotation concentrate produced in the rougher cell is adjusted with a recycle solution stream and fed to a 3.0 m³ cleaner flotation cell. The tailing from the cleaner cell is recycled to rougher flotation and cleaner concentrate is pumped to a belt filter. Rougher tailing proceeds to the first of two 3.0 m³ scavenger cells.

The tailing slurry from scavenger flotation is delivered to a 5 m x 5 m surge tank. This tank is agitated for solids suspension and serves as the source of feed for the reducing leach autoclave.

The cleaner concentrate slurry is filtered on a 3 m² horizontal belt vacuum filter. The filtrate is recycled to the flotation steps for control of pulp density in each cell. The zinc concentrate cake is recommended as feed material for the zinc pressure leach circuit where further processing for eventual recovery of the contained metal values (primarily zinc) can be completed.

3.5.2 Operating Conditions and Expected ResultsRougher Flotation

Feed Slurry Solids Content (% by wt)	20
Number of Stages	1
Retention Time	10 min

Scavenger Flotation

Feed Slurry Solids Content (% by wt)	10
Number of Stages	2
Retention Time	12 min

Cleaner Flotation

Feed Slurry Solids Content (% by wt)	20
Number of Stages	1
Retention Time	5 min

Flotation Products

Analysis	Cleaner Concentrate	Oxidic Residue
Lead	1%	20%
Iron	10%	10%
Elemental Sulphur	20%	1%
Zinc	35%	7%

Distribution	Cleaner Concentrate	Oxidic Residue
Lead	2%	98%
Iron	30%	70%
Elemental Sulphur	90%	10%
Zinc	65%	35%

Flotation Concentrate Filtration

Type	Horizontal Belt
Filter Cake Moisture Content (% by wt)	<25
Filtration Rate	1000 kg/m ² -h

3.5.3 Process Control Highlights

The control of the pulp density in each flotation stage is important to ensure the desired separation. The efficiency of separation will also be influenced by the particle size of the zinc concentrate added as reagent in the neutralization-reduction step.

The process will incorporate the control of those parameters normally included in a flotation circuit, such as degree of aeration and agitation. Filtration of the zinc concentrate filter cake will supply solution for solids dilution in the cleaner cell.

3.6 REDUCING LEACH

The objectives of the reducing leach are:

- to extract the zinc, iron and copper from the zinc flotation tailing;
- to reprecipitate dissolved copper as copper sulphide;
- to convert lead in the solids to lead sulphate; and
- to convert all ferric iron to ferrous.

3.6.1 Process Description

The zinc flotation tailing slurry is mixed with elemental sulphur and then subjected to a reducing leach in a brick lined autoclave into which sulphur dioxide gas is injected.

After about four hours of leaching, the slurry is flashed to atmospheric pressure before advancing to the copper flotation step. Flash tank exit vapour contains sulphur dioxide and would be directed to the central stack for emission to the atmosphere. Plumbojarosite type minerals and zinc ferrites contained in the zinc flotation tailing are attacked in the reducing leach. Zinc, iron, and copper are dissolved but the presence of elemental sulphur causes the reprecipitation of copper as copper sulphide. Lead contained in plumbojarosite is converted to insoluble lead sulphate.

Elemental sulphur is prepared at the zinc pressure leaching plant before transfer to the oxide residue treatment plant. The tote bags containing sulphur are emptied into a 1 m diameter x 2.5 m high bin with a 60° cone bottom. A weigh belt then meters about 50 kg/h of sulphur into a 2 m x 2 m reducing leach feed tank. The sulphur is blended with the zinc flotation tailing at a ratio of 10 kilograms sulphur per tonne of oxide residue feed.

Lignosol solution prepared at the zinc pressure leach plant is received in a 1.5 m x 1.5 m holding tank for use in the oxide residue treatment circuit. A metering pump transfers the required volume of Lignosol into the reducing leach feed tank.

A rubber lined pump transfers the slurry from the reducing leach feed tank to an indirect heat exchanger which preheats the reducing leach feed slurry to about 100°C. The slurry then enters the first compartment of a six compartment autoclave having shell dimensions of about 3.2 m diameter and 15 m length from tan to tan. The autoclave is a lead and brick lined, carbon steel vessel rated for operation at 100°C and 100 kPa (gauge). An internal diameter of approximately 3.0 m and a length of 15 m tan to tan results in a working slurry volume of about 85 m³. The autoclave internals such as agitator shafts, impellers, dip pipes, and baffles are fabricated from titanium. Sulphur dioxide is sparged under the agitators which are fitted with an upper axial and lower radial flow impeller which provides for the intimate mixing of the residue, liquor, and SO₂.

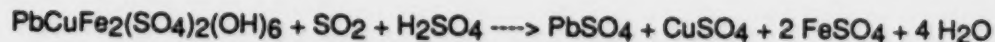
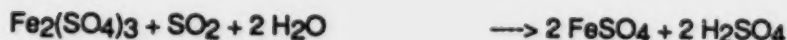
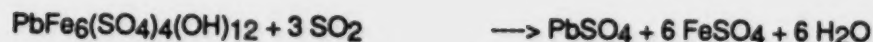
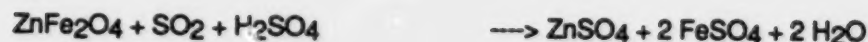
The reducing leach autoclave slurry will discharge through a 2 m diameter x 4 m high flash tank. Vapour from the flash tank is vented to the main stack for release of sulphur dioxide to the atmosphere. Slurry flows from the flash tank to a 3.5 m x 3.5 m surge tank which feeds the copper flotation circuit.

3.6.2 Operating Conditions and Expected Results

Reagents and Conditions

Reducing Agent	Sulphur Dioxide
Purity	100%
Utilization	60%
Average Sulphur Dioxide Requirement	170 kg/t oxide residue
Precipitating Agent	Sulphur
Average Sulphur Requirement	10 kg/t oxide residue
Calcium Lignosulphonate	5 g/t oxide residue
Pressure	100 kPa (gauge)
Temperature	100°C
Retention Time (nominal)	4 hours
Overall Conversions	
Zinc to Solution or Zinc Concentrate	92%
Iron to Solution	92%
Ferric Iron to Ferrous Iron	100%
Copper to Copper Sulphide	100%
Discharge Solution Composition	
Copper	0.01 g/L
Iron	37 g/L

Ferrous Iron	37 g/L
H ₂ SO ₄	5 g/L
Zinc	120 g/L

Overall Reactions3.6.3 Process Control Highlights

In the reducing leach, the leach conditions are controlled such that iron and zinc extractions are maximized. Net zinc extraction is significantly affected by the efficiency of the scavenging operation in the zinc flotation step. Any zinc sulphide reporting to the zinc flotation tailing is not attacked in a reducing leach and therefore has a negative impact on the zinc content of the copper concentrate product.

The elemental sulphur addition rate is set in proportion to the flow of residue into the autoclave. The scavenger flotation tailing flow is about equal to the oxide residue feed, therefore, the target weight ratios may be more conveniently set based on the oxide residue feed rate. In any case, sulphur addition would be optimized during commercial operations depending on the solution copper concentration. Sufficient calcium ligno-sulphonate would be added to wet the elemental sulphur.

The total pressure is controlled in the autoclave system and a bleed sufficient to remove inert compounds is maintained to give an appropriate sulphur dioxide partial pressure.

Careful control of the available process surges is of paramount importance for the commercial plant to achieve optimal consumption of reagents during the leaching steps. For this reason, large slurry storage tanks have been recommended ahead of the hot acid leach and the reducing leach process sections.

3.7 COPPER FLOTATION

The objective of the copper flotation step is to separate the reducing leach discharge solids into a copper rich concentrate and a lead rich residue. The concentrate is filtered and washed before transfer to the copper concentrate production circuit where it is blended with other feed for the copper smelter.

3.7.1 Process Description

The reducing leach residue is subjected to a rougher flotation followed by further upgrading of the rougher concentrate in a cleaner stage. Rougher tailing is treated in two scavenger flotation stages to minimize the sulphide and sulphur content in the oxidic residue which reports to the scavenger tailing. Scavenger tailing slurry is thickened before advancing to the lead flotation step. The zinc solution advances to an iron removal step.

The reducing leach discharge slurry, scavenger concentrate, and cleaner tailing are blended and then fed to a 3.0 m³ rougher flotation cell. The reagent Z200 is added as a collector. The pulp density of the flotation concentrate produced in the rougher cell is adjusted with a recycle solution stream and fed to a 3.0 m³ cleaner flotation cell. The tailing from the cleaner cell is recycled to rougher flotation and cleaner concentrate is collected in a 2.5 m x 2.5 m filter feed tank. Rougher tailing proceeds to the first of two 3.0 m³ scavenger cells.

The tailing slurry from scavenger flotation is combined with the filtrate from the copper flotation tailing thickener underflow filter and filtrate from the copper concentrate wash filter in a 2 m x 2 m thickener feed tank. The resulting slurry is flocculated and fed to a 10 m diameter thickener. Thickener overflow is pumped to a 5 m x 5 m surge tank from which the 100 to 125 g/L zinc liquor is advanced to the iron removal step. Underflow is sent to lead flotation.

The cleaner concentrate slurry is filtered on a 2 m² plate and frame filter where the cake is washed with process water and blown dry with air. The filtrate is advanced to the flotation tailing thickener feed tank. The copper concentrate cake is recommended for blending with other feed for the copper smelter where further processing for eventual recovery of the contained metal values (primarily copper) can be completed.

3.7.2 Operating Conditions and Expected Results

Rougher Flotation

Collector Type	Z200
Collector Dosage	350 g/t

Feed Slurry Solids Content (% by wt)	12
Number of Stages	1
Retention Time	10 min

Scavenger Flotation

Feed Slurry Solids Content (% by wt)	8
Number of Stages	2
Retention Time	10 min

Cleaner Flotation

Feed Slurry Solids Content (% by wt)	6
Number of Stages	1
Retention Time	5 min

Flotation Products

Analysis	Cleaner Concentrate	Oxidic Residue
Lead	0.2%	28%
Iron	0.7%	2.2%
Zinc	1.0%	1.5%
Copper	50%	0.3%

Distribution	Cleaner Concentrate	Oxidic Residue
Lead	0.1%	99%
Iron	2%	98%
Zinc	20%	80%
Copper	90%	10%

Flotation Concentrate Filtration

Type	Plate and Frame
Filter Cake Moisture Content (% by wt)	<10
Filtration Rate	1500 kg/m ² -h

Copper Flotation Tailing Thickener

Feed Slurry Solids Content (% by wt)	7
Flocculant Type	Percol 351
Flocculant Dosage	50 g/t
Thickener Underflow Solids	20%
Unit Area	1.0 m ² /t-d

3.7.3 Process Control Highlights

The control of the pulp density in each flotation stage is important to ensure the desired separation. To achieve a high grade copper concentrate, it is also necessary to separate all zinc sulphides from the slurry during the zinc flotation step.

The process will incorporate the control of those parameters normally included in a flotation circuit, such as the degrees of aeration and agitation. Washing of the copper concentrate filter cake will minimize entrainment of process solution which can contain deleterious impurities such as arsenic.

3.8 LEAD FLOTATION

The objective of the lead flotation step is to separate the gypsum and gangue minerals from the copper flotation tailing, thus concentrating lead sulphate into a high grade lead residue. The high grade lead residue is filtered and washed on a tower filter press to about 12% moisture before loading onto railcars. The lead residue can then be sold to a lead smelter.

3.8.1 Process Description

The copper flotation tailing thickener underflow is filtered and washed to minimize soluble zinc losses. The filter cake containing lead sulphate is then repulped and calcium hydrosulphide solution is added to activate the lead sulphate particles. This sulphidization process is assisted by increasing the slurry pH to about pH 9.5 with lime slurry. The activated residue is subjected to a rougher flotation followed by further upgrading of the rougher concentrate in a cleaner stage. Rougher tailing is treated in scavenger flotation to minimize the lead sulphate losses to the gypsum residue which reports to the scavenger tailing.

The copper flotation tailing thickener underflow is the feed for the lead flotation step. The thickener underflow slurry at 20% by weight solids is filtered on a vacuum drum filter where the cake is washed with process water. The filtrate is recycled to the thickener feed tank.

The washed lead-gypsum residue is dropped into a 4 m x 4 m repulp tank where the pulp density of the residue is adjusted with a recycle solution stream. The slurry pH is adjusted to about pH 9.5 with lime, and a sulphidizing reagent, calcium hydrosulphide, is added to activate the lead sulphate particles in the residue. The resulting slurry is then blended with scavenger concentrate and cleaner tailing and fed to a 3.0 m³ rougher flotation cell. A xanthate, such as Aero 350, is added as a collector. The pulp density of the flotation concentrate produced in the rougher cell is adjusted with a recycle solution

stream and fed to a 3.0 m³ cleaner flotation cell. The tailing from the cleaner cell is recycled to rougher flotation and cleaner concentrate is collected in a filter feed tank. Rougher tailing proceeds to the first of two 3.0 m³ scavenger cells.

The cleaner concentrate slurry is filtered on a 12.5 m² tower filter press of the Hoesch or Larox design where the cake is dewatered to a moisture content of about 12% by weight. The filtrate is recycled to the flotation steps for control of pulp density in each cell. The lead residue cake is transferred via conveyor belt to a rail car. The lead residue is then sold to a custom lead smelter for recovery of the contained lead.

The lead flotation tailing is transferred to the Gypsum Precipitation circuit and combined with gypsum residues produced by other operations at the HBM&S refinery. All of the residues are then stockpiled at a new tailings pond.

3.8.2 Operating Conditions and Expected Results

Copper Flotation Tailing Filter

Type	Vacuum Drum
Feed Solids	20%
Filter Cake Solids	70%
Wash Water Addition	2 Displacements
Washing Efficiency	95%
Filter Area	0.3 m ² /t-d

Rougher Flotation

Activator Type	Calcium Hydrosulphide
Activator Dosage	2000 g/t
pH Adjustment Reagent	Lime
Flotation pH	9.5
Frother Type	Dowfroth 200
Frother Dosage	250 g/t
Collector Type	Aero 350
Collector Dosage	2000 g/t
Feed Slurry Solids Content (% by wt)	15
Number of Stages	1
Retention Time	10 min

Scavenger Flotation

Feed Slurry Solids Content (% by wt)	12
Number of Stages	2
Retention Time	12 min

Cleaner Flotation

Feed Slurry Solids Content (% by wt)	5
Number of Stages	1
Retention Time	5 min

Flotation Products

Analysis	Cleaner Concentrate	Gypsum Residue
Pb	60%	2%
Distribution	Cleaner Concentrate	Gypsum Residue
Pb	95%	5%

Flotation Concentrate Filtration

Type	Tower Filter
Filter Cake Moisture Content	12%
Filtration Rate	0.5 m ² /t-d

3.8.3 Process Control Highlights

The process control of the circuit will incorporate those items that are normally needed for thickening, filtering, and flotation unit operations. In addition, pH control with lime slurry will be required to ensure that the lead sulphate particles are effectively activated by calcium hydrosulphide.

3.9 IRON REMOVAL

The objectives of the iron removal step are:

to neutralize acid and precipitate iron from the copper flotation tailing thickener overflow and produce an iron free zinc sulphate solution suitable for conventional purification and electrowinning processes;

to minimize zinc loss to the precipitated solids; and

to produce an iron removal precipitate amenable to separation from the zinc sulphate solution and to disposal in a tailings impoundment area.

3.9.1 Process Description

About 470 m³/d of solution containing about 5 g/L of sulphuric acid and the iron derived from the oxide residue leach circuit will be treated prior to conventional purification and electrowinning.

In the first stage of the iron removal circuit, solution from the copper flotation tailings thickener overflow surge tank is pumped into the first of three 3.5 m diameter x 4.0 m high agitated tanks arranged in cascade. Solids from the second stage of iron removal are also pumped into the first tank to dissolve zinc hydroxides entrained in the second stage solids. Zinc hydroxide sludge is added to the first, second, and (possibly) third tanks to neutralize acid and to maintain the solution at about pH 3.5. Oxygen is sparged into the tanks to oxidize ferrous iron in solution to the ferric state. The temperature is maintained at 85°C with steam to assure a reasonable rate of reaction. Slurry from the third tank overflows to a 10 m diameter thickener.

The thickener underflow slurry is pumped to a 2.5 m x 2.5 m tank and then to a 20 m² drum filter. The washed filter cake is repulped and discarded in a tailings impoundment area. The filtrate is pumped along with the first stage thickener overflow solution to the first tank of the second stage of iron removal, which consists of two 3.5 m diameter x 4.0 m high reaction tanks arranged in cascade followed by a clarifier. Slaked lime is added to the first tank to maintain a solution pH of 5.0 in the discharge of the second tank. Oxygen is sparged into both tanks to oxidize ferrous iron remaining in solution. The discharge from the second tank is treated in a 5 m diameter x 3 m high clarifier. The clarifier underflow is recycled to the first stage of iron removal. A portion of the clarifier overflow solution, iron free zinc sulphate solution, is recycled to the feed preparation section for repulping of the oxide residue; and the remainder is combined with solution produced in the main zinc plant iron removal section.

3.9.2 Operating Conditions and Expected Results

Reagents and Conditions

Temperature	85°C
Pressure	Atmospheric
Reactor Retention Time	80 minutes/tank
Zinc Sludge Requirement	550 kg/t oxide residue
Lime Requirement	9 kg/t oxide residue
Oxygen Gas (99.5% Pure)	90 kg/t oxide residue
Solids Production	520 kg/t oxide residue

Solids Analyses

	<u>First Stage</u>	<u>Second Stage</u>
Arsenic	4%	n.a.
Iron	22%	10%
Zinc	1%	40%

Solution Analyses

	<u>Feed</u>	<u>First Stage</u>	<u>Second Stage</u>
Arsenic	6 g/L	<0.1 g/L	<1 mg/L
Iron	34 g/L	<1 g/L	<20 mg/L
Zinc	115 g/L	140 g/L	140 g/L

Liquid-Solid SeparationFirst Stage Thickener

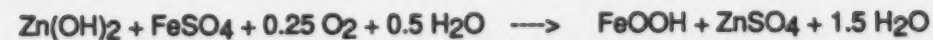
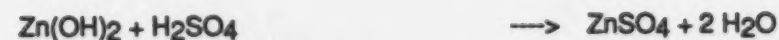
Feed Solids	6%
Underflow Solids	25%
Flocculant Type	Percol 351
Flocculant Dosage	100 g/t
Unit Area	1.0 m ² /t-d

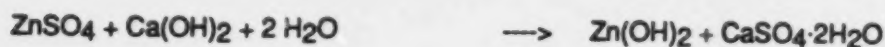
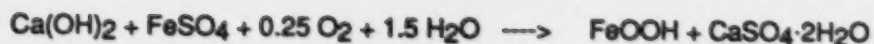
First Stage Filter

Type	Vacuum drum filter
Feed Solids	25%
Washed Filter Cake Solids	65%
Wash Ratio	2:1
Unit Area	0.3 m ² /t-d

Second Stage Clarifier

Feed Solids	1%
Underflow Solids	15%
Flocculant Type	Percol 156
Flocculant Dosage	1000 g/t
Unit Area	2.0 m ² /t-d
Overflow Clarity	50 mg/L

Overall Reactions



3.9.3 Process Control Highlights

A consistent feed solution is beneficial in maintaining low iron concentration in the iron removal product solution and low zinc content in the iron removal discharge solids. A reasonably large surge ahead of the iron removal circuit should be provided.

High concentrations of ferric iron in solution lead to the formation of slimy, difficult to filter precipitates; thus the concentration of ferric iron in the feed solution should be maintained at less than 1 g/L.

Ferric iron is rapidly hydrolyzed and precipitated at pH 3.5 in the first stage of iron removal, but oxidation of ferrous iron to the ferrous state requires good oxygen dispersion.

The first stage solution should be maintained below pH 4.0 to limit precipitation of zinc and copper hydroxides in the first stage and loss of these values to the discard iron cake. The solution should be maintained at a pH of at least 3.5 to avoid the formation of slimy precipitates.

The lime added to the second stage is to achieve a consistent pH of not greater than 5.

3.10 UTILITIES AND AUXILIARY FACILITIES

The oxide residue treatment process will require the following utilities:

- seal water supply;
- process water ;
- steam;
- plant and instrument air;
- vacuum supply; and
- electrical power.

The supply of the following reagents is to be provided:

- oxygen;
- sulphur;
- sulphur dioxide;
- lime slurry;

calcium lignosulphonate solution;
flotation reagents; and
miscellaneous chemicals.

3.11 PLANT EFFLUENTS

The main gaseous effluent is the sulphur dioxide vented together with water vapour from the reducing leach autoclave and flash tank. Other gaseous effluents include tank vent gases which are primarily air and water vapour. Tailing slurry will contain solids consisting of approximately 75 t/d of iron hydroxides and gypsum residues plus associated liquid effluent.

3.12 CAPITAL COST ESTIMATE

An order of magnitude capital cost estimate was prepared based on the flowsheet as described in Sections 3.2 to 3.9.

For the most part the equipment has been based on the preliminary evaluation of the Phase Ic test program. Where design data was unavailable the criteria is based on Sherritt's experience on similar unit operations. The costs applied to the equipment are derived from in-house data. Factors were applied to the equipment costs to assign costs for bulk commodities such as piping, electrical, instrumentation, etc. All costs are on a Western Canada location basis.

3.12.1 Battery Limits

The capital cost estimate is based on the supply of oxygen, steam, process and instrument air, vacuum, water, sulphur dioxide, zinc sludge, lime, and Lignosol at the oxide treatment plant battery limit. Zinc concentrate is received by conveyor belt and sulphur is received as powder in a tote bag. The upgraded lead residue would be shipped by rail car. No provision for on-site storage has been provided. The product copper concentrate is transferred to the existing copper flotation plant as a wet cake while the product zinc concentrate is pipelined as a 70% solids slurry to the main zinc plant.

3.12.2 Capital Cost Estimate

The order of magnitude capital cost of the oxide residue treatment facility in 1990 first quarter Canadian dollars for a Western Canada location is \$23 million.

	<u>Cost \$000 Canadian</u>
Oxide Residue Reclaim	600
Hot Acid Leach	2 600

Neutralization-Reduction	1 300
Zinc Flotation	1 200
Reducing Leach	4 600
Copper Flotation	1 600
Lead Flotation	2 000
* Iron Removal	2 500
Process Buildings	<u>2 000</u>
Total Installed Equipment Cost	18 400
Engineering, Procurement and Construction Management	3 300
Contingency	<u>3 300</u>
Total Project Cost	25 000

3.12.3 Exclusions From Capital Cost Estimate

The capital cost estimate is for a battery limit process plant only and excludes the following items:

offsites (site preparation, area lighting, fire loops, roads, parking, storm sewers, etc.);

sulphur dioxide production plant;

utilities plants;

main substation and power distribution;

oxide residue thaw shed and storage;

storage of upgraded lead residue for sale;

support facilities such as maintenance shops, laboratories, administrative offices, warehouses, etc.;

mobile equipment;

tailings ponds;

freight, insurance, sales tax and other duties for the purchased equipment;

land acquisition and other administrative costs;

technology licensing fees;

interest during construction;

working capital;

startup commissioning expense; and
escalation.

3.13 OPERATING COST ESTIMATE

The operating cost estimate was prepared based on the material balance included in Section 4, Process Chemistry, and outlined in Sections 3.2 to 3.9 above.

The costs are for treating 40 000 tonnes per year of oxide residue and are on a Western Canada location basis.

3.13.1 Operating Costs

Item	Unit	Unit Cost (\$/Unit)	Consumption (kg per t of Residue)	Annual Cost (\$)	\$ per t of Residue
Oxygen(99%)	t	120	180	864 000	21.60
SO ₂ Liquid	t	190	170	1 292 000	32.30
Limestone	t	100	400	1 600 000	40.00
Lime	t	400	20	320 000	8.00
Dowfroth 200	kg	25	0.048	48 000	1.20
Ca(HS) ₂	kg	10	0.4	160 000	4.00
Flocculant	kg	6	0.08	19 200	0.48
Lignosol	kg	1	0.005	200	0.01
Aero 350	kg	25	0.4	400 000	10.00
Filter Cloth and Miscellaneous	m ²	4	5 000*	20 000	0.50
Operating Labour	many year	40 000	20*	800 000	20.00
Misc. Operating Supplies	7% of Oper. Labour	-	-	56 000	1.40
Maintenance Labour	many year	40 000	12*	480 000	12.00
Steam, 35 psig	t	6	24 000*	144 000	3.60
Demineralized Water	m ³	1	2 400*	2 400	0.06
Cooling Water	m ³	0.02	0*	0	0.00
Power	MWH	35	200*	7 000	0.18
Instrument Air	Normal m ³	0.004	800 000*	3 200	0.08
Process Air	Normal m ³	0.004	800 000*	3 200	0.08
Maintenance Materials		-	-	<u>600 000</u>	<u>15.00</u>
TOTAL OPERATING COSTS				\$6 819 200	\$170.48

Pb Treatment Cost	lb of lead	0.22	10 198 349**	2 243 637	56.09
Cu Treatment Cost	lb of copper	0.18	1 206 029**	217 085	5.43
Zn Treatment Cost	lb of zinc	0.18	7 264 384**	1 307 589	32.69
GRAND TOTAL OPERATING COSTS				\$10 587 511	\$264.69

- * Annual requirement
- ** Annual recovered pounds

4.0 PROCESS CHEMISTRY

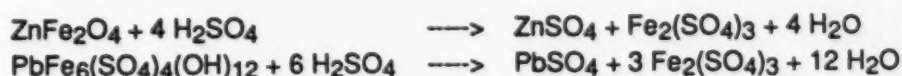
The process chosen for treatment of the HBM&S oxide residue involves many steps to allow for good separation of the metal values. A description of the composition of the feed material is given here, along with important chemical reactions taking place in the various process steps, and a detailed description of the deportment of individual elements in the proposed process.

4.1 FEED MINERALOGY

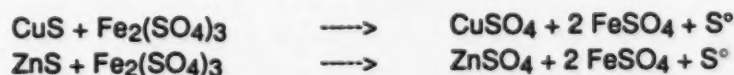
The mineralogy of the HBM&S zinc oxide fume leach residue has been described in some detail in the Phase Ia and Phase Ib reports. The principal minerals are anglesite (PbSO_4), a jarosite-like mineral, and zinc bearing oxides of the spinel group, some of which have the composition ZnFe_2O_4 . Other minerals include gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), sphalerite (ZnS), and a covellite-like copper sulphide. From the behaviour of copper in the process, it is apparent that about 20 to 30% of the copper is found as oxidic minerals, perhaps as beaverite, $\text{PbCuFe}_2(\text{SO}_4)_2(\text{OH})_6$. The residues obtained from reducing leaches of the oxide residue were found to contain zinc as sphalerite, gahnite (ZnAl_2O_4) and zinc silicate.

4.2 HOT ACID LEACH

A portion of the zinc ferrite and jarosite is digested with zinc spent electrolyte in the hot acid leach step, as outlined in the reactions below.



Ferric iron extracted into the solution reacts with copper sulphides to extract copper, as shown in below. Partial extraction of zinc from sphalerite also takes place.



4.3 NEUTRALIZATION-REDUCTION

Excess acid in the hot acid leach solution is neutralized with zinc sulphide under oxygen sparging conditions in the neutralization-reduction step.

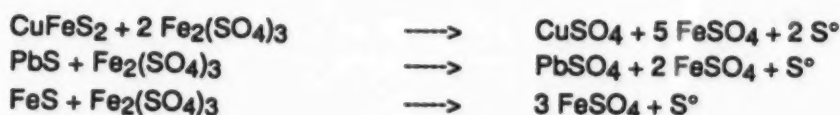


This reaction is impractically slow in the absence of a species which will facilitate oxygen transfer. One such species is dissolved iron, which is present in the hot acid leach

solution. Ferric iron reacts with sphalerite, as shown in the previous reaction, and acid is consumed according to the following reaction.



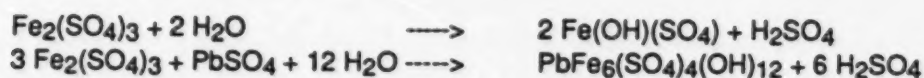
Other sulphide minerals in the zinc concentrate also react with ferric iron in the neutralization-reduction step.



A net reduction of ferric iron is allowed in the neutralization-reduction step, but the conditions are maintained sufficiently oxidizing to prevent precipitation of copper by metathesis, as shown below.

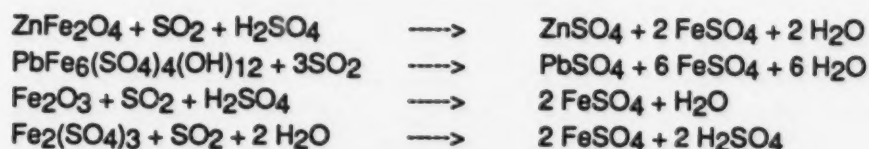


A low concentration of ferric iron in the solution is required as the acid concentration decreases, to limit hydrolysis and reprecipitation of iron, as given below.

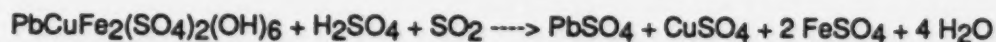


4.4 REDUCING LEACH

Iron bearing minerals in the zinc flotation tailing solids and ferric iron in the solution react with sulphur dioxide in the reducing leach, as given below.

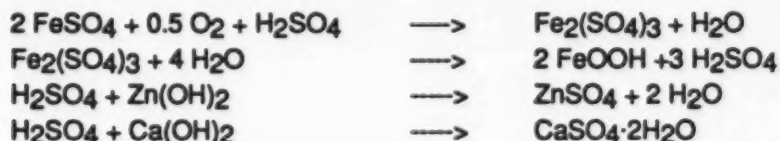


Copper in oxidic minerals such as beaverite is also initially extracted, as shown below. However, addition of elemental sulphur to the reducing leach promotes the precipitation of copper, and copper in both the feed solution and solids is converted to copper sulphide.

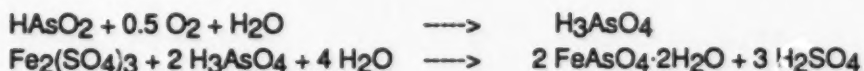


4.5 IRON REMOVAL

Iron in the reducing leach solution is virtually all in the ferrous state, and is oxidized and precipitated from solution as ferric oxide-hydroxide in the iron removal step. Acid in the feed solution, and acid generated during hydrolysis of iron, is neutralized with zinc hydroxide or slaked lime.



Arsenic in solution is oxidized to arsenate and precipitated from solution as ferric arsenate, as shown below. Other impurity elements such as germanium and fluoride also report in large part to the iron precipitate.



Copper and zinc in the solution may also be hydrolyzed as the pH is increased, and precipitate as hydroxides or basic sulphates.



4.6 ELEMENTAL DEPARTMENT

The detailed department of individual elements in the process is outlined in the following section. The distributions are based on analyses and flowrates from the continuous testwork, and batch test results for lead flotation and iron removal. The experimental material balance is given in Table 4.1, along with a normalized balance for processing all of the feed to the finished products, based on 100 kg of oxide residue. A lead tie was used in the normalization, and for balancing feed and product solids in the hot acid leach, neutralization-reduction, zinc flotation and reducing leach steps. An iron tie was used for the iron removal step. Analyses for all of the process streams are summarized in Tables 4.2 and 4.3. Weight distributions and accountabilities based on the normalized material balance and the analyses are given in Tables 4.4 to 4.7.

4.6.1 Antimony

The oxide residue sample treated in the current testwork contained 0.2% Sb. About one third of the antimony was extracted in the hot acid leach step, to give a solution containing 118 mg/L Sb. Antimony was largely reprecipitated in the neutralization-reduction and reducing leach steps, and reported principally to the flotation concentrates.

reduction and reducing leach steps, and reported principally to the flotation concentrates. The zinc, copper and lead concentrates contained 460, 4220 and 2660 g/t Sb, respectively. About 6% of the antimony was precipitated in the iron removal step and reported to the iron cake. The second stage iron removal solution contained <0.1 mg/L Sb.

4.6.2 Arsenic

The stockpiled oxide residue contains up to 5% As, although the present sample contained only 1.3% As. About 75% of the arsenic was extracted in the hot acid leach step to yield a solution containing 2.8 g/L As. About 20 % of the dissolved arsenic was precipitated in the neutralization-reduction step, but was redissolved in the reducing leach step. The feed solution to the iron removal step contained about 3 g/L As. Arsenic was effectively removed from solution in iron removal to yield a zinc sulphate solution containing <1 mg/L As. With filtering and washing of the flotation concentrates and return of the solutions to the oxide residue circuit, the distribution of arsenic would be about 80% to the iron cake, 8% to the zinc concentrate, 6% to the copper concentrate, 4% to the lead concentrate and < 1% to the lead tailing.

4.6.3 Cadmium

Cadmium deportment was similar to that of zinc, with about 35% extraction in the hot acid leach. The net cadmium extraction from the oxide residue after the reducing leach was about 70%. The iron free zinc sulphate solution contained 0.2 g/L Cd.

4.6.4 Calcium

The process solutions were all saturated with gypsum and contained about 0.5 g/L Ca. The oxide residue feed contained 6.4 % Ca, principally as gypsum. Gypsum was readily separated from copper sulphide and lead sulphate in the reducing leach residue by flotation. About 94% of the gypsum in the feed reported to the lead flotation tailing; 1% to the lead flotation concentrate; and about 5% to the copper flotation concentrate. Better rejection of calcium from the copper concentrate was achieved in batch tests carried out after the continuous testwork. The lead flotation tailing contained about 20% by weight calcium, or 85 to 90% gypsum.

Calcium was also added during the iron removal step, as either lime or zinc hydroxide sludge, and reported quantitatively to the precipitate. The first stage iron removal cake contained 14% Ca, again principally as gypsum.

4.6.5 Chloride

The oxide residue contained only 11 g/t Cl, and the principal source of chloride in the process was the zinc concentrate added during neutralization-reduction, which contained 50 g/t Cl.

There was no pickup of chloride to the solution in the hot acid leach, which contained 8 mg/L Cl. Extraction from the zinc concentrate was about 38%, and the neutralization-reduction solution contained 12 mg/L Cl. Chloride was further extracted in the reducing leach, and the solution concentration increased to between 14 and 17 mg/L Cl. The concentration of chloride in solution was unaffected in the iron removal process, and thus the iron free zinc sulphate solution also contained 17 mg/L Cl, representing a net pickup of 8 mg/L Cl.

4.6.6 Copper

Copper was added with the oxide residue, which contained 1.61% Cu, the zinc concentrate, which contained 1.15% Cu, and the zinc hydroxide sludge, which contained 0.2% Cu. Copper extraction in the hot acid leach step was between 65 and 70% to yield a solution containing 3.2 g/L Cu. There was a small net extraction of copper in the neutralization-reduction step, and the neutralization-reduction solution contained 3.5 g/L Cu. Copper extracted in this step originated from the zinc concentrate, which contained 23% of the copper in the total feed. About 20% of the total copper, unleached copper from the zinc concentrate added during neutralization-reduction, reported to the cleaner concentrate solids in the subsequent zinc flotation step. The solution component of the zinc flotation cleaner concentrate contained 13% of the copper in the feed. If this solution was recovered, and returned to the oxide residue circuit, eventual recovery of copper to the copper flotation concentrate would be about 65%. This would represent about 87% of the copper in the oxide residue feed.

About 9% of the copper reported to the copper flotation tailing solids in the continuous testwork, and most of this eventually reported to the lead flotation concentrate, which contained about 1% Cu by weight.

Although the copper concentrate contained only 16% Cu by weight in the continuous flotation test, subsequent batch tests demonstrated that a concentrate containing 50% Cu could be obtained. Production of a high grade copper concentrate requires efficient flotation of zinc sulphide in the zinc flotation step and treatment of fresh reducing leach discharge slurry in copper flotation.

Precipitation of copper in the reducing leach was essentially quantitative, with typically only 0.01 g/L Cu in the discharge solution. Since copper is thought to catalyze the

oxidation and hydrolysis of iron during iron removal, it may be necessary to bypass the reducing leach with a small solution stream to ensure there is sufficient copper in the feed to iron removal. This slip stream could be a portion of the zinc cleaner concentrate filtrate and wash solution.

The feed solution to iron removal contained 0.24 g/L Cu and precipitation of iron was rapid. About half of the copper was precipitated in iron removal, with the first stage solution containing 0.09 to 0.19 g/L Cu. This precipitated copper would be lost to the iron cake. The copper concentration decreased to between 0.05 and 0.13 g/L in the second stage of iron removal, but this copper could be recovered by recycle of the second stage solids to the first stage. Copper in the iron free solution can be recovered in the zinc dust purification process. The minimum copper concentration required in iron removal should be better quantified to allow for a better determination of the expected copper loss in this circuit.

4.6.7 Fluoride

The oxide residue contained 121 g/t F, of which 67% was extracted into solution in the hot acid leach step. The net fluoride extraction was 80 to 85% after the reducing leach, with 36 mg/L F in the reducing leach solution. Most of the extracted fluoride was reprecipitated in iron removal, with 6 mg/L F remaining in the iron free zinc sulphate solution. No net pickup of F to the solution is expected if this iron removal method is used in the process.

4.6.8. Germanium

The stockpiled residue contains about 100 g/t Ge and this represents a significant percentage of the contained metals value. However, the oxide residue contains high concentrations of iron and arsenic, and germanium behaves in a similar manner to these elements in the process; rendering concentration or recovery of the germanium very difficult. Germanium recovery was not a process objective for the current phase of work.

The oxide residue sample treated in the continuous testwork contained 88 g/t Ge, of which about 70% was extracted in the hot acid leach step to yield a solution containing 16 mg/L Ge. There was a small increase in the net extraction in the reducing leach to yield a solution containing 18 mg/L Ge. The extracted germanium was reprecipitated in the iron removal step, with the iron free zinc sulphate solution containing only 1 mg/L Ge. Of the unleached germanium, about half reported to the lead concentrate, which contained 85 g/t Ge.

4.6.9. Iron

Iron is the major contaminant in the oxide residue, and it is necessary to initially extract iron into solution to allow for high zinc recovery and the production of a high grade lead concentrate. Since the concentration of iron in the oxide residue is comparable to that of zinc, the concentration of zinc in the solution is limited by the capacity of the solution to maintain a high concentration of iron during the leaching steps.

About 35% of the iron was extracted into the solution during the continuous hot acid leach test. The hot acid leach solution contained 15 to 20 g/L Fe, most of which was in the ferric state. The zinc concentrate added during neutralization-reduction contained leachable iron, and the total concentration of iron in the neutralization-reduction solution was about 3 g/L higher than in the hot acid leach solution. Most of the iron was in the ferrous state in the neutralization-reduction solution. The net iron extraction from the oxide residue increased to 92% in the reducing leach step and the discharge solution contained 40 to 45 g/L Fe, virtually all in the ferrous state.

With recycle of filtrate and wash solutions from the flotation concentrates, virtually all of the extracted iron would report to the first stage iron removal cake. This cake contained 21 to 22% iron in the iron removal tests, where zinc hydroxide sludge was used as the neutralizing agent. If lime were used, dilution with gypsum would result in solids containing about 17% Fe. The second stage iron removal solution, the feed to conventional purification and electrowinning steps, contained less than 1 mg/L Fe.

4.6.10 Lead

The oxide residue feed sample contained 13.7% lead in the continuous testwork. Lead was found principally as lead sulphate and plumbojarosite. Lead sulphate remained unattacked in the leaching steps, while plumbojarosite was largely converted to lead sulphate. The zinc concentrate added during neutralization-reduction contained 0.3% Pb by weight, representing about 1% of the total lead feed.

About 10% of the lead in the feed reported to the zinc flotation concentrate in the continuous testwork. This lead could ultimately be recovered if the zinc flotation concentrate were to be fed to a zinc pressure leach circuit, but it is unlikely that this much lead would report to the zinc concentrate in commercial practice, as described below. Of the remaining lead, about 5% reported to the copper concentrate, 4% reported to the lead tailing, and 81% reported to the lead flotation concentrate, which contained 60% lead by weight.

Rejection of lead to the zinc and copper flotation tailing fractions was significantly better in batch tests than in the continuous testwork, and it is expected that less than 3% of

the lead would report to the zinc concentrate and less than 1% would report to the copper concentrate in practice. Recovery of lead to the lead concentrate would be expected to be between 90 and 95%.

4.6.11 Silica

The oxide residue contained about 5% SiO_2 . About half of the silica eventually reported to the lead flotation concentrate, with the remainder split almost equally between the zinc concentrate, copper concentrate, and lead tailing solids. The feed to the iron removal step contained 0.3 g/L SiO_2 , most of which reported to the first stage iron removal cake.

4.6.12 Silver

Recovery of silver is important to the overall process economics. The oxide residue sample treated in the continuous testwork contained 163 g/t Ag, while the zinc concentrate added during neutralization-reduction contained 41 g/t Ag. If all of the silver contained in the zinc concentrate is considered to report to the zinc flotation concentrate following neutralization-reduction, the distribution of silver contained in the oxide residue can be calculated by difference. About 74% of the oxide residue silver reported to the zinc flotation concentrate; 11% to the copper concentrate; 10% to the lead concentrate; and 5% the lead tailing. With the exception of silver in the lead tailing, all of this silver is recoverable.

4.6.13 Sulphur

A sulphur balance is provided for the solids only. The oxide residue contained about 9% sulphur, of which 7.5% was sulphate sulphur and the balance was sulphide sulphur. Sulphur was also added in the form of sulphides with the zinc concentrate, and as elemental sulphur in the reducing leach step. The zinc flotation concentrate accounted for 68% of the sulphur in the product solids. This concentrate contained 9% elemental sulphur on average, but the percentage of elemental sulphur in this fraction would be expected to increase with more efficient use of zinc concentrate in the neutralization-reduction step, as described in Section 8. The copper concentrate contained nearly 10% of the sulphur in the feed solids, principally as sulphide sulphur and elemental sulphur, while the lead concentrate and lead tailing contained 3% and 20% respectively, principally as sulphate.

The iron cake contained about 13% by weight sulphur as sulphate. This sulphur was precipitated from the solution during iron removal, principally as gypsum.

4.6.14 Zinc

The stockpiled oxide residue contains between 10 and 15% zinc. The sample treated in the continuous testwork contained 11.5% Zn. Zinc in the oxide residue feed is distributed between numerous compounds, including zinc ferrite, zinc sulphide and relatively inert phases such as zinc silicate and aluminate. The process chosen is designed for the dissolution of oxidic compounds such as zinc ferrite, and recovery of sulphidic zinc by flotation. Because of the high concentration of iron in the oxide residue, the concentration of zinc in the solution cannot conveniently be increased to a very high level without an additional source of zinc, such as zinc concentrate in the neutralization-reduction step and zinc hydroxide sludge in the iron removal step.

Zinc extraction in the hot acid leach step was about 35%, and the hot acid leach discharge solution contained 63 g/L Zn on average. Owing to dilution with the large quantity of water contained with the oxide residue feed, the concentration of zinc in the hot acid leach discharge solution was virtually the same as that of the spent electrolyte feed to the hot acid leach. The concentration increased to 81 g/L Zn in the neutralization-reduction step, and to 90.5 g/L Zn in the reducing leach step. The reducing leach solution also contained 42 g/L Fe. Zinc hydroxide sludge was used for neutralization in the iron removal tests and the second stage iron removal solution contained 114 g/L Zn.

Distribution of zinc in the total feed to the various process streams is outlined in Table 4.7. The distribution of the zinc contained in the oxide residue to the products can be calculated by assuming that 26% of the zinc in the hot acid leach residue is recoverable as zinc sulphide in the zinc flotation step, as indicated by Batch Test 4, Section 9, and that all of the excess zinc concentrate added during neutralization-reduction is recovered with the zinc flotation concentrate. Accordingly, about 10% of the zinc in the oxide residue feed reported to the zinc flotation concentrate in the continuous testwork; 9% to the copper concentrate; 5% to the lead concentrate; 1% to the lead tailing; 4.4% to the iron cake solids; 2.5% to the entrained iron cake solution; and 68% to the product zinc sulphate solution.

Batch tests carried out after completion of the continuous testwork demonstrated that better recovery of zinc sulphide could be achieved in zinc flotation, and the copper flotation concentrate would be expected to contain only about 1.5% of the zinc in the oxide residue feed, compared with 9% in the continuous test. The zinc flotation concentrate would therefore be expected to contain about 17.5% of the zinc in the oxide residue feed, and total recovery of zinc to the zinc flotation concentrate and the zinc sulphate solution would increase to about 85%.

Table 4.1 Material Balance

Component	Experimental		Normalized	
	Weight kg	Volume L	Weight kg	Volume L
Hot Acid Leach				
Oxide Residue Solids	195.0		1000.0	
Oxide Residue Solution		226.9		1163.6
Fortified Spent Electrolyte		521.8		2675.9
Discharge Solids	153.8		788.7	
Discharge Solution		703.0		3605.1
Neutralization-Reduction				
Feed Slurry Solids	124.2		788.7	
Feed Slurry Solution		572.2		3605.1
Zinc Concentrate Solids	77.4		491.5	
Zinc Concentrate Solution		33.2		210.8
Discharge Solids	172.1		1092.6	
Discharge Solution		550.6		3496.5
Zinc Flotation				
Feed Slurry Solids	156.6		1223.9	
Feed Slurry Solution		469.4		3496.5
Synthetic Solution		50.4		375.4
Cleaner Concentrate Solids	64.6		504.9	
Cleaner Concentrate Solution		122.6		958.2
Scavenger Tailing Solids	92.0		719.0	
Scavenger Tailing Solution		377.4		2949.6
Reducing Leach				
Feed Slurry Solids	69.9		714.9	
Feed Slurry Solution		286.7		2949.6
Elemental Sulphur	0.9		8.8	
Sulphur Dioxide	12.8		131.3	
Discharge Solids	48.6		497.4	
Discharge Solution		295.7		3026.1
Copper Flotation				
Feed Slurry Solids	50.7		562.9	
Feed Slurry Solution		270.8		3026.1
Cleaner Concentrate Solids	7.7		85.5	
Cleaner Concentrate Solution		41.8		467.1
Scavenger Tailing Solids	43.0		477.4	
Scavenger Tailing Solution		229.0		2559.0
Lead Flotation				
Feed			477.4	
Cleaner Concentrate			192.8	
Scavenger Tailing			284.6	
Iron Removal				
Feed Solution				2837.0
Zinc Hydroxide Sludge			552.0	
Iron Cake Solids			521.5	
Iron Cake Solution				280.8
Product Solution				4050.5

Table 4.2 Summary of Analyses (Antimony to Fluoride)

Component	Analysis, % or g/L						
	Sb*	As	Cd	Ca	Cl*	Cu	F*
Hot Acid Leach							
Oxide Residue Solids	1550	1.33	0.12	6.35	11	1.61	121
Oxide Residue Solution			0.05	0.51	10	0.07	2
Fortified Spent Electrolyte				0.15	9		3
Discharge Solids	780	0.46	0.093	7.3	14	0.68	50
Discharge Solution	118	2.8	0.13	0.63	8	3.19	27
Neutralization-Reduction							
Feed Slurry Solids	780	0.46	0.093	7.3	14	0.68	50
Feed Slurry Solution	118	2.8	0.13	0.63	8	3.19	27
Zinc Concentrate Solids	12		0.11	0.01	50	1.15	26
Zinc Concentrate Solution			0.007		26	0.02	3
Discharge Solids	890	0.46	0.10	5.79	20	0.78	37
Discharge Solution	76	2.56	0.17	0.54	12	3.51	30
Zinc Flotation							
Feed Slurry Solids	890	0.38	0.10	4.59	20	0.82	37
Feed Slurry Solution	76	2.56	0.17	0.54	12	3.51	30
Synthetic Solution						3.94	
Cleaner Concentrate Solids	460	0.2	0.13		37	1.06	21
Cleaner Concentrate Solution	69	2.31	0.15	0.49	11	3.55	27
Scavenger Tailing Solids	1080	0.54	0.07	8.68	19	0.59	41
Scavenger Tailing Solution	69	2.31	0.15	0.49	11	3.55	27
Reducing Leach							
Feed Slurry Solids	1080	0.53	0.07	8.7	19	0.6	41
Feed Slurry Solution	69	2.2	0.15	0.49	11	3.62	27
Discharge Solids	1410	0.25	0.07	9.22	5	3.13	39
Discharge Solution	8	2.91	0.20	0.47	14	0.01	36
Copper Flotation							
Feed Slurry Solids	1246	0.07	0.06	10.9	5	2.85	34
Feed Slurry Solution	8	2.91	0.20	0.47	14	0.01	36
Cleaner Concentrate Solids	4220	0.97	0.09	3.8	12	16.0	36
Cleaner Concentrate Solution	8	2.91	0.20	0.47	14	0.01	36
Scavenger Tailing Solids	770	0.09	0.06	11.2	5	0.43	12
Scavenger Tailing Solution	8	2.91	0.20	0.47	14	0.01	36
Lead Flotation							
Feed	770	0.09	0.06	11.2	5	0.43	12
Cleaner Concentrate	2660	0.28	0.10	0.4	5	1.12	37
Scavenger Tailing	140	0.02	0.01	22.7	7	0.07	22
Iron Removal							
Feed Solution	21	2.19	0.14	0.47	17	0.24	29
Zinc Hydroxide Sludge				11.8	21	0.20	11
Iron Cake Solids	140	1.36	0.01	14.2	<6	0.3	44
Iron Cake Solution			0.02	0.05	2		1
Product Solution	0.1	<0.001	0.16	0.51	17	0.05	6

* Analysis g/t or mg/L

Table 4.3 Summary of Analyses (Germanium to Zinc)

Component	Analysis, % or g/L						
	Ge*	Fe	Pb	SiO ₂	Ag*	S	Zn
Hot Acid Leach							
Oxide Residue Solids	88	14.5	13.7	4.71	163	9.0	11.5
Oxide Residue Solution							24.3
Fortified Spent Electrolyte				0.03			64.4
Discharge Solids	33	11.6	17.4	4.94	201		10.5
Discharge Solution	16	14.7					63.1
Neutralization-Reduction							
Feed Slurry Solids	33	11.6	17.4	4.94	201		10.5
Feed Slurry Solution	16	14.7					63.1
Zinc Concentrate Solids		11.4	0.3	0.45	41	32.8	48.8
Zinc Concentrate Solution		0.5					4.7
Discharge Solids	26	11.2	12.7	4.03	118		19.8
Discharge Solution	17	18.9					81
Zinc Flotation							
Feed Slurry Solids	26	10.4	11.3	3.02	118	22.3	24.2
Feed Slurry Solution	17	18.9					81
Synthetic Solution		21					79.1
Cleaner Concentrate Solids	17	9.6	2.8	1.17	271	37.2	43.8
Cleaner Concentrate Solution	15	19.1					80.8
Scavenger Tailing Solids	31	10.8	17.3	4.65	61	11.2	7.3
Scavenger Tailing Solution	15	19.1					80.8
Reducing Leach							
Feed Slurry Solids	31	10.7	17.4	4.62	61	11.2	7.0
Feed Slurry Solution	15	21.1		0.13			81.6
Discharge Solids	33	2.2	25.0	7.08	82		3.3
Discharge Solution	18	42.4		0.33			90.5
Copper Flotation							
Feed Slurry Solids	29	1.2	22.1	7.37	72	15.1	2.7
Feed Slurry Solution	18	42.4		0.33			90.5
Cleaner Concentrate Solids	59	4.2	7.4	9.56	210	30.5	13.3
Cleaner Concentrate Solution	18	42.4		0.33			90.5
Scavenger Tailing Solids	33	0.8	25.5	7.38	56	12.8	1.3
Scavenger Tailing Solution	18	42.4		0.33			90.5
Lead Flotation							
Feed	33	0.8	25.5	7.38	56	12.8	1.3
Cleaner Concentrate	85	1.4	60.0	10.3	83	3.9	3.2
Scavenger Tailing	7	0.4	2.1	3.2	28	19.8	0.5
Iron Removal							
Feed Solution	14	38.2		0.33			81.5
Zinc Hydroxide Sludge	4	0.1				12.8	26.7
Iron Cake Solids	63	20.9		0.6	3	12.5	0.8
Iron Cake Solution							8.2
Product Solution	<1	<0.001					91.9

* Analysis g/t or mg/L

Table 4.4 Weight Distributions (Antimony to Fluoride)

Component	Weight, kg						
	Sb*	As	Cd	Ca	Cl*	Cu	F*
Hot Acid Leach							
Oxide Residue Solids	1550	13.3	1.2	63.5	11.0	16.1	121
Oxide Residue Solution	0	0.0	0.1	0.6	11.1	0.1	2.3
Fortified Spent Electrolyte	0	0.0	0.0	0.4	23.0	0.0	8.3
Discharge Solids	615	3.7	0.7	57.6	11.0	5.4	39.4
Discharge Solution	425	10.1	0.5	2.3	28.8	11.5	97.3
Neutralization-Reduction							
Feed Slurry Solids	615	3.7	0.7	57.6	11.0	5.4	39.4
Feed Slurry Solution	425	10.1	0.5	2.3	28.8	11.5	97.3
Zinc Concentrate Solids	6	0.0	0.5	0.0	24.6	5.7	12.8
Zinc Concentrate Solution	0	0.0	0.0	0.0	5.5	0.0	0.6
Discharge Solids	972	5.1	1.1	63.3	21.9	8.5	40.4
Discharge Solution	266	9.0	0.6	1.9	42.0	12.3	104.9
Zinc Flotation							
Feed Slurry Solids	1089	4.7	1.2	56.2	24.5	10.0	45.3
Feed Slurry Solution	266	9.0	0.6	1.9	42.0	12.3	104.9
Synthetic Solution	0	0.0	0.0	0.0	0.0	1.5	0.0
Cleaner Concentrate Solids	232	1.0	0.7	0.0	18.7	5.4	10.6
Cleaner Concentrate Solution	66	2.2	0.1	0.5	10.4	3.4	26.0
Scavenger Tailing Solids	776	3.9	0.5	62.4	13.7	4.2	29.5
Scavenger Tailing Solution	196	6.8	0.5	1.4	32.0	10.5	79.9
Reducing Leach							
Feed Slurry Solids	772	3.8	0.5	62.2	13.6	4.3	29.3
Feed Slurry Solution	202	6.5	0.4	1.4	31.9	10.7	79.9
Discharge Solids	701	1.2	0.3	45.9	2.5	15.6	19.4
Discharge Solution	24	8.8	0.6	1.4	42.4	0.0	108.9
Copper Flotation							
Feed Slurry Solids	701	0.4	0.3	61.4	2.8	16.0	19.4
Feed Slurry Solution	24	8.8	0.6	1.4	42.4	0.0	108.9
Cleaner Concentrate Solids	361	0.8	0.1	3.2	1.0	13.7	3.1
Cleaner Concentrate Solution	4	1.4	0.1	0.2	6.5	0.0	16.8
Scavenger Tailing Solids	368	0.4	0.3	53.5	2.4	2.1	5.7
Scavenger Tailing Solution							
Lead Flotation							
Feed	368	0.4	0.3	53.5	2.4	2.1	5.7
Cleaner Concentrate	513	0.5	0.2	0.8	1.0	2.2	7.1
Scavenger Tailing	40	0.1	0.0	64.6	2.0	0.2	6.3
Iron Removal							
Feed Solution	60	6.2	0.4	1.3	48.2	0.7	82.3
Zinc Hydroxide Sludge	4	0.0	0.0	65.1	11.6	1.1	6.1
Iron Cake Solids	73	7.1	0.1	74.1	0.0	1.6	22.9
Iron Cake Solution	0	0.0	0.0	0.0	0.5	0.0	0.2
Product Solution	0	0.0	0.6	2.1	68.9	0.2	24.3

* Weight in g

Table 4.5 Weight Distributions (Germanium to Zinc)

Component	Weight, kg						
	Ge*	Fe	Pb	SiO ₂	Ag*	S	Zn
Hot Acid Leach							
Oxide Residue Solids	88	145.0	137.0	47.1	163	89.9	115.0
Oxide Residue Solution	0	0.0	0.0	0.0	0		28.3
Fortified Spent Electrolyte	0	0.0	0.0	0.1	0		172.3
Discharge Solids	26	91.3	137.0	39.0	158		82.6
Discharge Solution	58	53.0	0.0	0.0	0		227.5
Neutralization-Reduction							
Feed Slurry Solids	26	91.3	137.0	39.0	158		82.6
Feed Slurry Solution	58	53.0	0.0	0.0	0		227.5
Zinc Concentrate Solids	0	56.0	1.3	2.2	20	161.2	239.9
Zinc Concentrate Solution	0	0.1	0.0	0.0	0		1.0
Discharge Solids	28	122.9	138.3	44.0	129		216.1
Discharge Solution	59	66.1	0.0	0.0	0		283.2
Zinc Flotation							
Feed Slurry Solids	32	127.3	138.3	37.0	144	272.9	296.2
Feed Slurry Solution	59	66.1	0.0	0.0	0		283.2
Synthetic Solution	0	7.9	0.0	0.0	0		29.7
Cleaner Concentrate Solids	9	48.7	13.9	5.9	137	187.8	221.1
Cleaner Concentrate Solution	15	18.3	0.0	0.0	0		77.4
Scavenger Tailing Solids	22	77.7	124.4	33.4	44	80.5	52.7
Scavenger Tailing Solution	45	56.3	0.0	0.0	0		238.4
Reducing Leach							
Feed Slurry Solids	22	76.5	124.4	33.0	44	80.1	50.3
Feed Slurry Solution	45	62.2	0.0	0.4	0		240.7
Discharge Solids	16	10.7	124.4	35.2	41		16.6
Discharge Solution	54	128.2	0.0	1.0	0		273.9
Copper Flotation							
Feed Slurry Solids	16	6.6	124.4	41.5	41	85.0	15.2
Feed Slurry Solution	54	128.2	0.0	1.0	0		273.9
Cleaner Concentrate Solids	5	3.5	6.3	8.2	18	26.1	11.4
Cleaner Concentrate Solution	8	19.8	0.0	0.2	0		42.3
Scavenger Tailing Solids	16	4.0	121.7	35.2	27	61.1	6.3
Scavenger Tailing Solution	46	108.4	0.0	0.8	0		231.6
Lead Flotation							
Feed	16	4.0	121.7	35.2	27	61.1	6.3
Cleaner Concentrate	16	2.7	115.7	19.9	16	7.5	6.1
Scavenger Tailing	2	1.1	6.1	9.1	8	56.4	1.5
Iron Removal							
Feed Solution	40	108.4	0.0	0.9	0	0.0	231.2
Zinc Hydroxide Sludge	2	0.6	0.0	0.0	0	70.7	147.4
Iron Cake Solids	33	109.0	0.0	3.1	2	65.2	4.1
Iron Cake Solution	0	0.0	0.0	0.0	0	0.0	2.3
Product Solution	4	0.0	0.0	0.0	0	0.0	372.2

* Weight in g

Table 4.6 Overall Elemental Balance and Accountability

Component	Accountability, % *							
	HAL	NR	ZF	RL	CF	LF	IR	Overall
Antimony	67	118	94	74	104	150	115	83
Arsenic	103	102	102	98	109	139	114	99
Cadmium	96	97	97	101	103	77	177	106
Calcium	93	109	111	74	93	122	114	112
Chloride	88	91	112	99	101	124	116	126
Copper	104	92	99	104	98	115	100	109
Fluoride	104	97	97	117	92	234	54	78
Germanium	95	105	100	105	106	117	88	102
Iron	100	94	100	100	101	97	100	97
Lead	100	100	100	100	103	100		103
Silica	83	107	106	108	104	82	334	94
Silver	97	72	125	94	110	90		98
Sulphur **			98		103	105	92	107
Zinc	98	91	97	100	101	121	100	101

* HAL = Hot Acid Leach; NR = Neutralization-Reduction; ZF = Zinc Flotation;
 RL = Reducing Leach; CF = Copper Flotation; LF = Lead Flotation; IR = Iron Removal

** Solids only

Table 4.7 Product Distribution

Product	Distribution, %						
	Sb	As	Cd	Ca	Cl	Cu	F
Zinc Concentrate Solids	18.0	7.7	34.5	0.0	17.2	20.1	9.0
Zinc Concentrate Solution*	5.1	16.9	7.7	0.3	9.5	12.8	22.1
Copper Concentrate Solids	28.0	6.3	4.1	2.2	0.9	51.5	2.6
Copper Concentrate Solution*	0.3	10.4	4.9	0.2	6.0	0.0	14.3
Lead Concentrate Solids	39.8	4.1	10.1	0.5	0.9	8.1	6.1
Lead Tailing Solids	3.1	0.4	1.5	44.4	1.8	0.7	5.3
Iron Cake Solids	5.7	54.1	2.7	50.9	0.0	5.9	19.6
Iron Cake Solution	0.0	0.0	0.2	0.0	0.4	0.0	0.1
Zinc Sulphate Solution	0.0	0.0	34.1	1.4	63.2	0.8	20.7
Total	100	100	100	100	100	100	100

Product	Distribution, %						
	Ge	Fe	Pb	SiO ₂	Ag	S**	Zn
Zinc Concentrate Solids	9.3	24.0	9.8	12.8	75.9	67.6	29.9
Zinc Concentrate Solution*	16.0	9.0	0.0	0.0	0.0	0.0	10.5
Copper Concentrate Solids	5.5	1.7	4.5	17.6	10.0	9.4	1.5
Copper Concentrate Solution*	9.1	9.7	0.0	0.3	0.0	0.0	5.7
Lead Concentrate Solids	17.8	1.3	81.4	42.9	8.9	2.7	0.8
Lead Tailing Solids	2.3	0.6	4.3	19.7	4.4	20.3	0.2
Iron Cake Solids	35.6	53.6	0.0	6.8	0.9	0.0	0.6
Iron Cake Solution	0.0	0.0	0.0	0.0	0.0	0.0	0.3
Zinc Sulphate Solution	4.4	0.0	0.0	0.0	0.0	0.0	50.4
Total	100	100	100	100	100	100	100

* These solutions would be returned to the process in an integrated operation

** Distribution of sulphur originating from feed solids only

5.0 FEED MATERIALS

Two shipments of materials were received from HBM&S. The type and quantity of material received in each shipment is listed below.

Date of Arrival	Material	Weight, kg * (Volume, L)	Moisture Content %
March 15, 1989	Oxide Residue	8670	46.0
	Zinc Hydroxide Sludge	1736	n.a.
May 12, 1989	Purified Neutral Leach Solution	(300)	-
	Zinc Concentrate	170	8.4

* Weight of moist cake

n.a. = not analyzed

5.1 OXIDE RESIDUE

The shipment of oxide fume leach residue received from HBM&S was of fresh residue product which had been sampled on February 23 and March 2, 1989. The moist cake was blended with demineralized water and purified neutral leach solution to give a slurry containing about 45% solids for feed to the continuous hot acid leach circuit. Average analyses for the feed samples obtained during the continuous hot acid leach test are summarized in the table below. More detailed analyses are provided in Table 5.1.

Analysis, %							
As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn
1.3	0.1	6.4	1.6	14.5	13.7	4.7	11.5

The initial batch tests were carried out with a sample of oxide residue obtained from HBM&S on December 5, 1987 during Phase Ib of the study. Analyses for this sample, and other samples previously tested by Sherritt are given in Table 5.1.

The oxide residue sample treated in the continuous testwork in July and August, 1989 was lower in arsenic and lead, and higher in iron content than the samples tested in December 1987.

The oxide residue consisted primarily of very fine particles. The size analysis of the sample used in the continuous testwork is given overleaf. The value given for the +1000 μ m fraction, 0.1%, was determined from the weight of solids collected on the feed screen during the continuous testwork.

Oxide Residue	Sieve Opening, μm				
	1000	125	88	61	44
Cumulative Weight % Passing	99.9	99.3	98.7	96.9	94.1

These solids contained some wood chips and other tramp material, but consisted mostly of black chunks of about 1 mm in diameter. The chemical analysis for the oversize material is given below.

Analysis, %									
As	Cd	Ca	Cu	Fe	Pb	SiO ₂	S(SO ₄)	S	Zn
0.19	0.21	0.61	2.98	10.1	2.13	3.94	1.5	14.8	36.0

5.2 ZINC CONCENTRATE

The zinc concentrate received from HBM&S was coarse, 45% greater than 44 microns, and was reground in a ball mill before use in the continuous neutralization-reduction test. The size analysis for the reground concentrate is given below.

HBM&S Zinc Concentrate	Sieve Opening, μm			
	125	88	61	44
Cumulative Weight % Passing	100	99.7	97.6	90.2

The reground concentrate was diluted with demineralized water to a 70% solids slurry, pulp density 2100 g/L, for feed to the neutralization-reduction circuit. The chemical analyses of the reground concentrate and slurry water are provided in Table 5.2.

An Australian zinc concentrate was used in the batch neutralization-reduction tests because HBM&S concentrate was not available at the time. This concentrate was 80% less than 44 microns and 99% less than 125 microns in size. The chemical analyses of both concentrates are summarized in the table below.

Concentrate	Analysis, %					
	Cd	Cu	Fe	Pb	S	Zn
HBM&S	0.11	1.15	11.4	0.27	32.8	48.8
Australian (Batch Tests)	0.16	0.13	8.58	2.73	33.9	48.0

5.3 PURIFIED NEUTRAL LEACH SOLUTION

Purified neutral leach solution from HBM&S was used to prepare the fortified spent electrolyte feed to the hot acid leach, and to repulp the oxide residue solids for feed to the hot acid-leach. Analyses for the purified neutral leach solution, the fortified zinc spent electrolyte and the repulp solution are given in Table 5.3.

5.4 ELEMENTAL SULPHUR

Flowers of sulphur (Fisher Scientific) was used to precipitate copper in the reducing leach. Sulphur was added batchwise to each pail of feed along with 0.1 mL of 300 g/L Lignosol solution as a wetting agent (750 g Lignosol per tonne elemental sulphur, or about 5 g/t oxide residue).

5.5 SULPHUR DIOXIDE

Sulphur dioxide (Matheson) was applied to the vapour space in the reducing leach to digest iron bearing minerals and to precipitate copper from solution. The gas was obtained in cylinders containing 18 kg SO₂, and was of reagent grade, 99.98% purity.

5.6 ZINC HYDROXIDE SLUDGE

Zinc hydroxide sludge was obtained from HBM&S for use as a neutralizing agent in the iron removal tests. The sludge was used as a 25% solids slurry and as a 43% solids cake. Analyses for the sludge solids and repulp solution are given in Table 5.4.

5.7 LIME

Processed lime was slaked to produce a 200 g/L CaO slurry for feed to the second stage of the iron removal tests. The lime slurry was also used to produce a calcium hydrosulphide solution for lead flotation. Analysis of the lime is provided below.

Reagent	Analysis, %		
	Ca	CO ₂	Mg
Lime	65.6	1.6	0.52

5.8 SODIUM SULPHIDE

Sodium sulphide (BDH) was used to sulphidize lead sulphate in lead flotation tests. The salt was obtained as the nonahydrate, and was dissolved in about 1 mL of demineralized water for addition to the flotation cell.

5.9 CALCIUM HYDROSULPHIDE

Calcium hydrosulphide was also used to sulphidize lead sulphate for lead flotation. A calcium hydrosulphide solution was prepared by diluting a 200 g/L CaO slurry to about 20 g/L CaO and sparging H₂S into the slurry until all of the lime dissolved. The resulting solution was deep yellow in colour and contained 12.9 g/L Ca.

5.10 POTASSIUM AMYL XANTHATE

Potassium amyl xanthate, as the reagent Aero 350 (Cyanamid) was used as a collector in lead flotation. The pale yellow solid was dissolved in demineralized water to a 1% solution for addition to the flotation cell.

5.11 HEXADECYL AMMONIUM CHLORIDE

Hexadecyl ammonium chloride was used as a flotation reagent for recovery of a lead sulphate flotation concentrate. This reagent was prepared by dissolving 2 g of hexadecylamine (Aldrich) in 200 mL of diethyl ether and bubbling HCl gas (Matheson) into the solution. The insoluble primary ammonium salt was immediately produced, and was recovered by filtration. The product was white and soapy in texture, and was added directly to the flotation cell as a solid.

Table 5.1 HBM&S Zinc Oxide Fume Leach Residue Analyses

	Sample			
	March 25, 1986	December 7, 1989	March 15, 1989	HBM&S*
Analysis, %				
Al	1.54	n.a.	n.a.	n.a.
Sb	0.36	n.a.	0.155	0.66
As	3.18	3.06	1.33	5.5
Bi	0.05	n.a.	n.a.	n.a.
Cd	0.19	0.16	0.12	0.15
Ca	6.31	6.18	6.35	n.a.
Cu	1.63	1.44	1.61	2.2
Fe	11.6	10.9	14.5	11.8
Pb	16.1	15.9	13.7	16.6
Mg	0.10	n.a.	n.a.	n.a.
Mn	0.05	n.a.	n.a.	n.a.
SiO ₂	3.34	3.96	4.71	n.a.
S(SO ₄)	8.7	7.80	7.50	n.a.
S	9.5	8.54	8.99	n.a.
Zn	9.72	8.94	11.5	14.1
Analysis, g/t				
Cl	n.a.	n.a.	11	n.a.
F	160	95	121	50
Ge	78	54	88	100
Au	2.4	2.3	n.a.	2.1
Ag	123	105	163	115
Specific Gravity	n.a.	n.a.	3.97	n.a.

* Typical analysis provided by HBM&S for the stockpile

n.a. = not analyzed

Table 5.2 HBM&S Zinc Concentrate Slurry Analyses

	Solids As Received	Solids Reground	Slurry Water
Analysis, % or g/L			
As	n.a.	<0.02	n.a.
Cd	0.11	0.11	0.007
Ca	0.08	0.01	n.a.
Cu	0.98	1.15	0.020
Fe	10.4	11.4	0.538
Pb	0.35	0.27	n.a.
Mg	0.16	n.a.	n.a.
Mn	0.13	n.a.	n.a.
SiO ₂	n.a.	0.45	n.a.
S(SO ₄)	0.1	<0.01	n.a.
S	33.9	32.8	n.a.
Zn	48.0	48.8	4.73
Analysis, g/t or mg/L			
Sb	n.a.	12	<0.02
Cl	n.a.	50	26
F	n.a.	26	3
Ge	n.a.	<0.5	<1
Ag	n.a.	41	n.a.
Specific Gravity	4.01	n.a.	n.a.

n.a. = not analyzed

Table 5.3 Feed Solution Analyses

	HBM&S Purified Neutral Leach Solution	Hot Acid Leach Fortified Spent Electrolyte	Hot Acid Leach Feed Slurry Solution
Analysis, g/L			
As	n.a.	<0.02	<0.02
Cd	<0.005	<0.005	0.05
Ca	0.38	0.15	0.51
Cu	<0.005	<0.005	0.07
Fe	<0.005	<0.005	0.01
Mg	4.62	n.a.	n.a.
Mn	1.50	n.a.	n.a.
K	0.10	n.a.	n.a.
SiO ₂	0.07	n.a.	n.a.
Na	0.30	n.a.	n.a.
H ₂ SO ₄	n.a.	1.52	n.a.
Zn	168	64.4	24.3
Analysis, mg/L			
Sb	0.05	n.a.	0.03
Cl	22.5	n.a.	9.5
F	8	n.a.	2
Ge	<1	n.a.	<1
Specific Gravity	1.40	n.a.	n.a.

n.a. = not analyzed

Table 5.4 Zinc Hydroxide Sludge Analyses

	Solids	Solution
Analysis, %		
Cd	0.07	n.a.
Ca	11.8	n.a.
Cu	0.20	n.a.
Fe	0.10	n.a.
Mg	0.20	n.a.
Mn	0.11	n.a.
S(SO ₄)	12.8	n.a.
Zn	26.7	n.a.
Analysis, g/t or mg/L		
Sb	7	<0.02
Cl	21	4.5
F	11	3
Ge	4.1	<1
Ag	2	n.a.

n.a. = not analyzed

6.0 EQUIPMENT AND PROCEDURES

The test program consisted of a combination of batch and continuous tests for the hot acid leach, neutralization-reduction, zinc flotation, reducing leach and copper flotation operations. Lead flotation and iron removal tests were carried out in a batch mode only. The continuous hot acid leach and neutralization-reduction tests were carried out concurrently while all other testwork was carried out in isolation.

6.1 DATA RECORDING

Batch and continuous test data, including operating parameters, control analyses and observations were recorded in activity books. These books also contained detailed operating and sampling procedures and instructions.

6.2 ANALYTICAL

Solution ferrous and acid analyses were conducted immediately after the samples were obtained, in the test laboratory. These control analyses allowed for quick adjustment of the process parameters. Detailed solids and solution analyses were carried out by Sherritt's Analytical Services Department.

6.2.1. Control Analyses

Solutions were analyzed for ferrous iron by titration with potassium dichromate. Free sulphuric acid was determined by titration with sodium carbonate to a pH of 3.5.

6.2.2 Sherritt Analyses

Solutions and solids were analyzed for metals using inductively coupled plasma (ICP) spectroscopy. The elements analyzed by ICP included arsenic, cadmium, calcium, copper, iron, lead, magnesium, manganese, silicon and zinc. Low concentrations of arsenic were determined by a wet collection method followed by ICP spectroscopy. Silver in the solids and copper and iron in the second stage iron removal solution were determined by atomic absorption spectroscopy. Antimony and germanium were determined by combined ICP mass spectroscopy. Chloride and fluoride were determined using specific ion electrodes. Sulphate sulphur was analyzed by conducting a sodium carbonate leach followed by barium sulphate precipitation. Elemental sulphur was determined by CS₂ extraction; and total sulphur by combustion.

6.3 HOT ACID LEACH

6.3.1 Batch Tests

Batch hot acid leach tests were carried out in an open stainless steel beaker. Heat was applied with a hot plate; and agitation with an overhead stirrer equipped with a marine impeller.

6.3.2 Continuous Test

The continuous hot acid leach test circuit is shown in Figure 6.1, and the equipment parameters are given in Table 6.1. The circuit consisted of a train of four agitated stainless steel tanks arranged in cascade, each with an operating volume of 30 L, an agitated slurry feed tank, a solution feed tank, and a water feed tank and distribution system. All of the tanks were equipped with Plexiglas covers. Marine impellers were used for agitation in the four reaction tanks, and in the slurry feed tank. HBM&S oxide residue was pumped as a 45% solids slurry to the first tank of the train of reaction tanks. Fortified zinc spent electrolyte was pumped separately to the same tank. Water was pumped equally to all four tanks to limit evaporation loss. Heat was applied with steam coils in the first and third reaction tanks, and with electric heating tapes on the second and fourth tanks. Overflow slurry was pumped to the neutralization-reduction circuit feed tank.

6.4 NEUTRALIZATION-REDUCTION

6.4.1 Batch Tests

Batch neutralization-reduction tests were carried out in a 4L autoclave equipped with baffles, dual impellers, gas sparge tube, thermocouple well, gas vent tube and discharge dip tube. Agitation was achieved with 4 blade axial over 4 blade radial impellers operating at a tip speed of 4.8 m/s. Oxygen was sparged into the slurry below the bottom impeller, with vent gas being continually withdrawn through a condenser and a rotameter. Concentrate slurry was charged into the autoclave by removing the condenser and injecting the slurry through the vent line with a pressurized bomb. The autoclave was externally heated by a natural gas flame and externally cooled by a water spray.

6.4.2 Continuous Test

The continuous neutralization-reduction test circuit is shown in Figure 6.2, and the equipment parameters are given in Table 6.1. The circuit consisted of a surge tank to accept slurry from the hot acid leach circuit, a train of five agitated stainless steel tanks arranged in cascade, each with an operating volume of 13.5 L, an agitated zinc concentrate slurry feed tank, and a water feed tank and distribution system. All of the tanks were equipped with Plexiglas covers and water was pumped equally to the five reaction

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tanks to make up evaporative losses. The first three reaction tanks were equipped with 4 blade axial over 6 blade radial impellers; and the last two with dual axial impellers. Heat was applied with steam coils in the first and third tanks, and with heating tapes for the remaining reaction tanks.

Hot acid leach slurry was pumped to the first reaction tank, and zinc concentrate slurry was pumped to the first and the third and/or fourth tanks. Oxygen was sparged into the first three or four tanks, with the gas inlet below the bottom impeller. Overflow slurry from the last tank was collected in pails.

6.5 FLOTATION

6.5.1 Batch Tests

Batch zinc, copper and lead flotation tests were conducted with a Denver Model D-12 laboratory flotation machine. Agitation was generally maintained at 1500 rev/min, with manual air regulation to maintain a stable froth.

6.5.2 Continuous Tests

Continuous zinc and copper flotation tests were carried out with continuous neutralization-reduction and reducing leach slurries, respectively. Circuits for these two flotation operations are shown in Figures 6.3 and 6.4. Equipment parameters are given in Table 6.2.

6.5.2.1 Continuous Zinc Flotation

Feed slurry was heated in a 200 L tank equipped with baffles, a steam coil and a 22.9 cm diameter marine impeller with a variable speed drive. Agitation was sufficient to maintain good solids suspension. Slurry was fed from the bottom of the feed tank to the rougher cell (Cell 2) of a four cell Denver "Sub-A" No. 7 flotation unit. Rougher concentrate overflowed into a launder, was diluted with synthetic cleaner concentrate filtrate, and was pumped to the cleaner cell (Cell 1). Cleaner concentrate was collected in pails. Cleaner tailing slurry proceeded to the rougher cell and rougher tailing slurry proceeded to the scavenger cells (Cells 3 and 4). The scavenger concentrates were collected in one launder and pumped together to the rougher cell. Scavenger tailing slurry was collected in a surge tank, where it was blended before distributing to pails.

6.5.2.2 Continuous Copper Flotation

Operation of the copper flotation circuit was similar to that of the zinc flotation circuit, except that a flotation reagent was required for copper flotation, and the tailing slurry was thickened.

The copper flotation feed slurry was treated with Z200 in a small agitated conditioning tank prior to being pumped to the rougher cell. The retention time in the premix tank was 1 minute. The Z200 was added continuously to maintain a rate of 350 g/t of feed solids.

Copper flotation tailing was thickened with Percol 351 in a Plexiglas thickener equipped with rakes. The flocculant was added as a 0.5 g/L solution. The flocculant solution was diluted with thickener overflow solution, and two thirds of the diluted solution was mixed with the feed slurry in a premix tank located directly over the thickener feed well. Flocculated slurry overflowed into the feed well. The remaining third of the diluted flocculant solution was fed separately, about halfway down the feed well.

A portion of the thickener overflow solution was recycled to the rougher concentrate launder to dilute the feed to the cleaner cell. The remainder was collected in a drum. The thickener underflow was collected in pails.

6.6 REDUCING LEACH

6.6.1 Batch Tests

Batch reducing leach tests were carried out in a 4 L titanium-clad autoclave equipped with baffles, dual impellers, thermocouple well, discharge dip tube and vapour inlet tube. Agitation was achieved with dual 4 blade axial impellers operating at a tip speed of 4.8 m/s. The autoclave was externally heated by a natural gas flame and externally cooled by a water spray.

All feed solids and solutions were charged to the autoclave before sealing the vessel. Once at temperature, air was vented from the vessel and sulphur dioxide was applied to the vapour space to maintain a constant pressure.

6.6.2 Continuous Test

Continuous zinc flotation tailing slurry was treated in the continuous reducing leach test. The continuous reducing leach circuit is shown in Figure 6.5 and the equipment parameters are given in Table 6.3.

Leaching was conducted in a six compartment, six agitator, horizontal autoclave. Each compartment of the autoclave was equipped with a thermocouple well, gas inlet, sample dip tube, baffles and a central agitator. The agitator shafts in the first five compartments were fitted with two 7.6 cm diameter axial impellers. The single impeller in the last compartment was an axial turbine. Slurry transfer between compartments was via downcomer tubes. Agitation at 1170 rev/min provided a tip speed of 4.7 m/s. Heating was

by natural gas flames located under each compartment. The nominal operating volume of the autoclave was 30 L. The body and internals of the autoclave were fabricated from titanium.

Elemental sulphur and Lignosol were blended with each pail of feed before addition to the agitated feed tank, which was placed on a weigh scale for determination of the feed rate. Slurry from the feed tank was fed continuously with a peristaltic pump to the first compartment of the autoclave. Sulphur dioxide was introduced to the vapour space in the first five compartments, to maintain a constant pressure. The sulphur dioxide cylinder was positioned on a weigh scale for determination of the feed rate. Slurry was continuously discharged from the last compartment of the autoclave to maintain a level in this compartment. The discharge passed into a Plexiglas cylinder to permit disengaging of vapour from the slurry which was pumped from the bottom of the cylinder into pails. A constant slurry level was manually maintained in the cylinder by visual inspection. The vapour phase, principally unreacted sulphur dioxide, was recycled to the first compartment of the autoclave with a peristaltic pump.

Initially the autoclave was discharged through a water cooled coil. Flashing of the discharge slurry proved to be essential in maintaining solution tenors and the cooling coil was removed halfway through the test.

6.7 IRON REMOVAL

Copper flotation tailing thickener overflow was treated with zinc hydroxide sludge and lime under oxygen sparging conditions to neutralize acid and precipitate iron in the iron removal tests. The batch tests were carried out in a 10.5 cm diameter glass cylinder, 29 cm in height. The vessel was equipped with baffles, dual radial impellers, a gas inlet tube and a Plexiglas cover. The vessel was externally heated by an electric tape. Agitation was achieved with dual 4 blade radial impellers operating at a tip speed of 5.2 m/s. Oxygen was sparged into the vessel below the lower impeller. Zinc hydroxide sludge or lime was added to maintain a constant pH.

After completion of the first stage of iron removal, the slurry was filtered, and the solution returned to the vessel for treatment in the second stage.

Table 6.1 Continuous Hot Acid Leach and Neutralization-Reduction Equipment Parameters

Circuit	Hot Acid Leach	Neutralization-Reduction
<u>Reaction Tanks</u>	HAL1 to HAL4	NR1 to NR5
Material	Type 316 Stainless Steel	Type 316 Stainless Steel
Inside Diameter	33 cm	26.5 cm
Slurry Height	34 cm	29 cm
Freeboard	11 cm	19 cm
Working Volume	30 L per tank	13.5 L per tank *
Baffles	Yes	Yes
Agitation Geometry	Marine	Axial over Radial (NR1 to NR3) Dual Axial (NR4 and NR5)
Impeller Diameter	14 cm	10 cm
Tip Speed	5.0 m/s	5.1 to 5.5 m/s
<u>Slurry Feed Tank</u>		
Material	Nalgene	Nalgene
Working Volume	25 L	25 L
Baffles	Yes	Yes
Agitation Geometry	Marine	Axial
Impeller Diameter	19 cm	14 cm
Tip Speed	5.0 m/s	3.5 m/s

* Ungassed slurry volume. The actual volume occupied by the gassed slurry is between 15 and 16 L.

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Table 6.2 Continuous Flotation Equipment Parameters

<u>Feed Tank</u>	
Material	Nalgene
Agitation Geometry	Marine
Impeller Diameter	23 cm
Tip Speed	Variable to maintain suspension
Volume	200 L nominal
Temperature Control	Steam coil
<u>Flotation Unit</u>	
Material	Stainless Steel
Type	Sub-aeration
Make, Model	Denver, "Sub-A" No. 7
Cells	Four, of equal volume
Working Volume	94 L total
<u>Thickener (Copper Flotation)</u>	
Material	Plexiglas
Diameter	20 cm
Cross Sectional Area	0.0314 m ²
Volume	48 L
Rake Type	Picket Fence
Rake Speed	2 rev/min

Table 6.3 Continuous Reducing Leach Equipment Parameters

<u>Autoclave</u> Material Working Volume Agitation Geometry Impeller Diameters Tip Speed	Titanium 30 L Dual axial * 7.6 cm 4.7 m/s
<u>Slurry-Vapour Separation Chamber</u> Material Diameter Cross Sectional Area Volume Slurry Volume	Plexiglas 10 cm 0.0079 m ² 8 L 2 L

* Single axial impeller in the last compartment

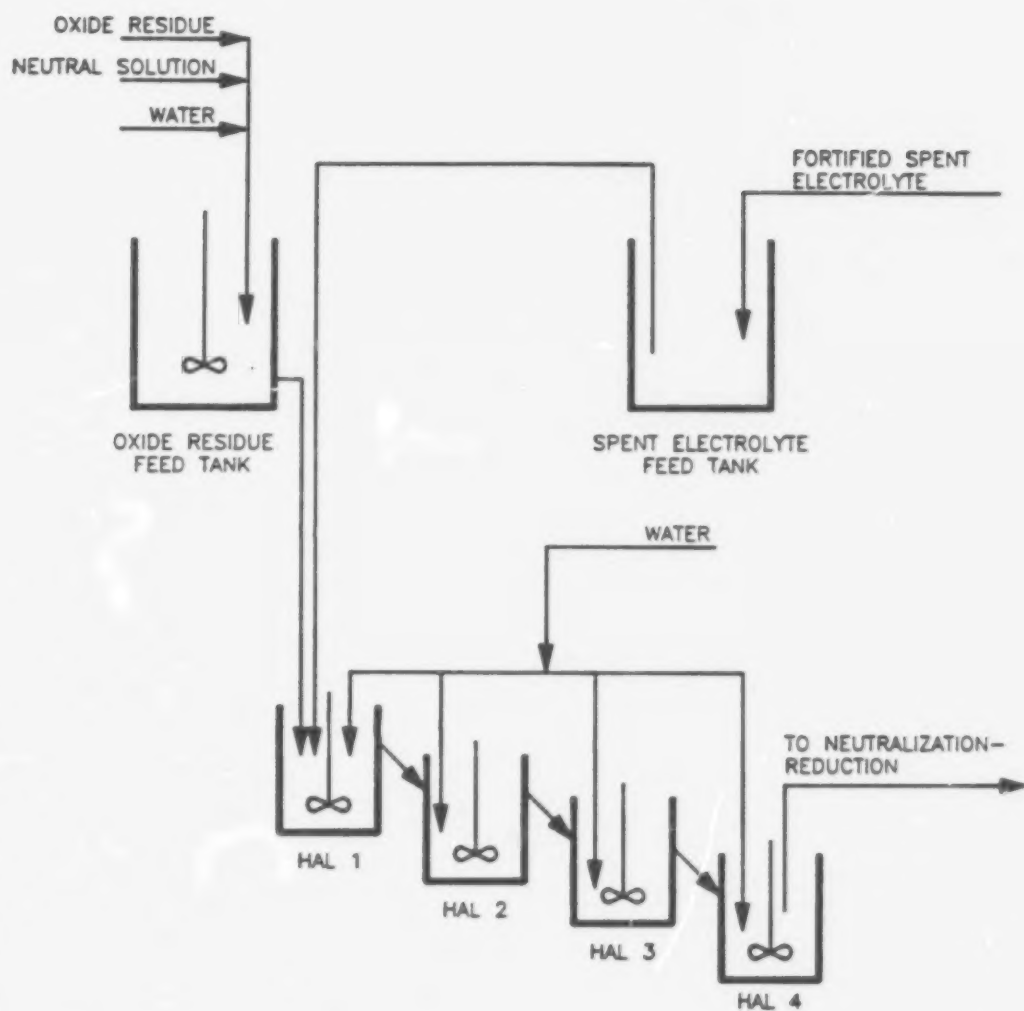


Figure 6.1 Continuous Hot Acid Leach Equipment Flowsheet

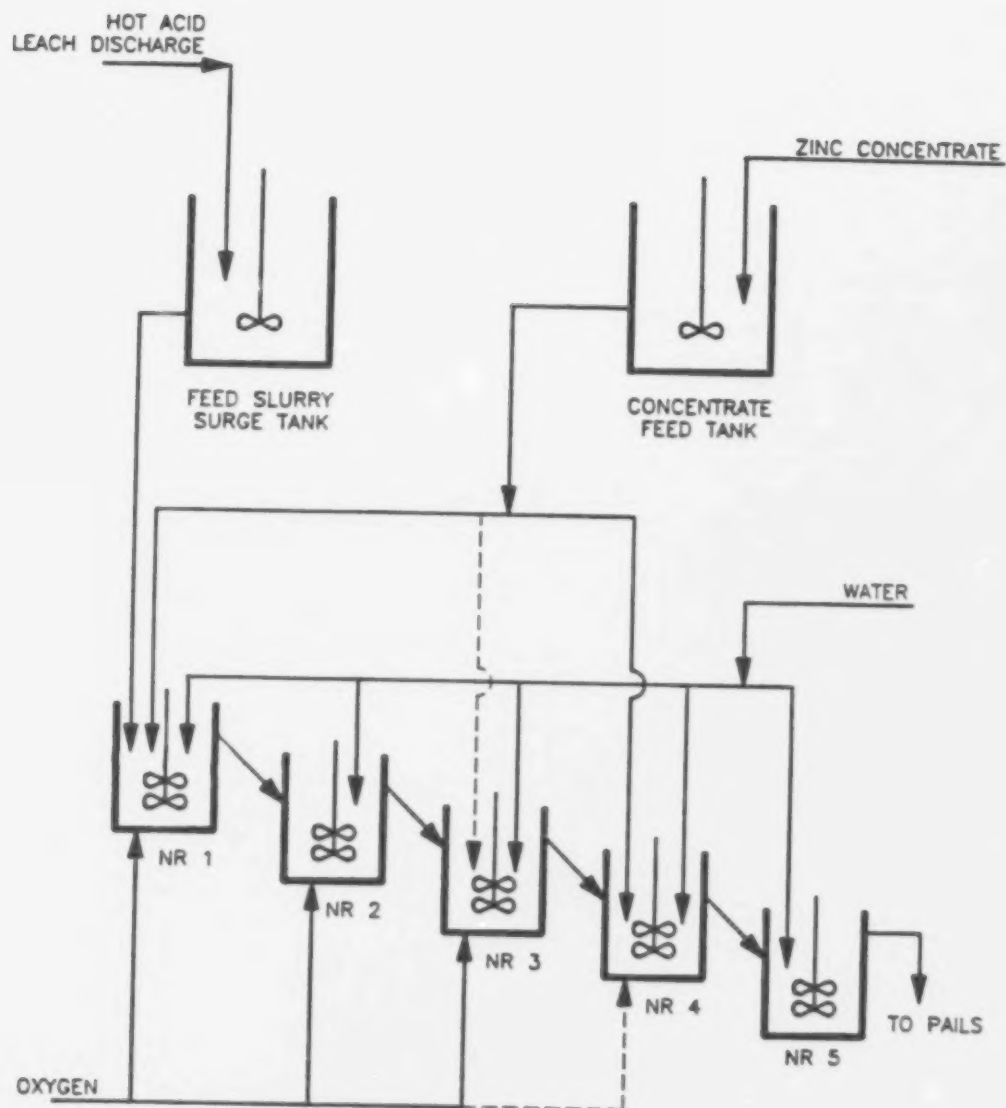


Figure 6.2 Continuous Neutralization-Reduction Equipment Flowsheet

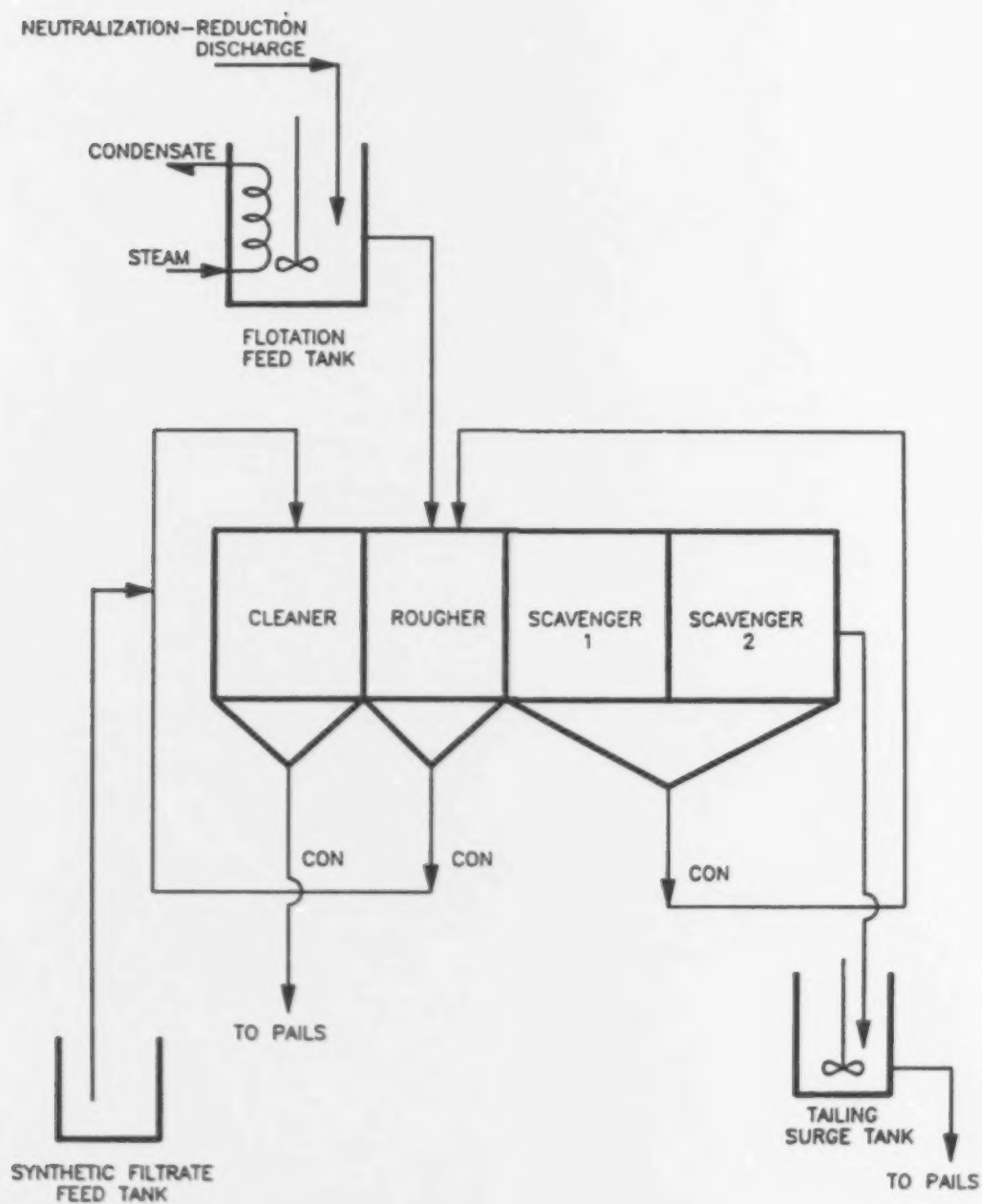


Figure 6.3 Continuous Zinc Flotation Equipment Flowsheet

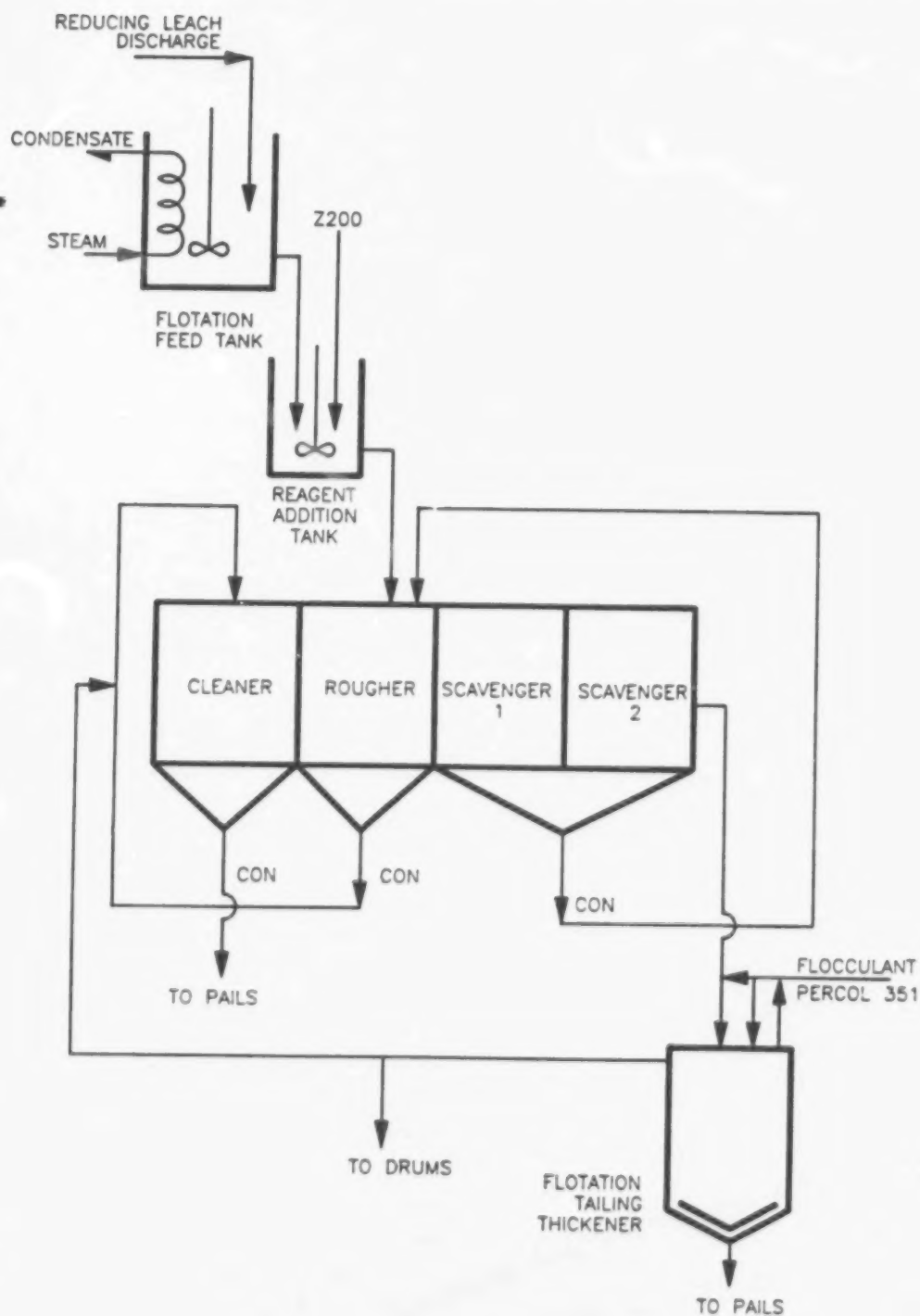


Figure 6.4 Continuous Copper Flotation Equipment Flowsheet

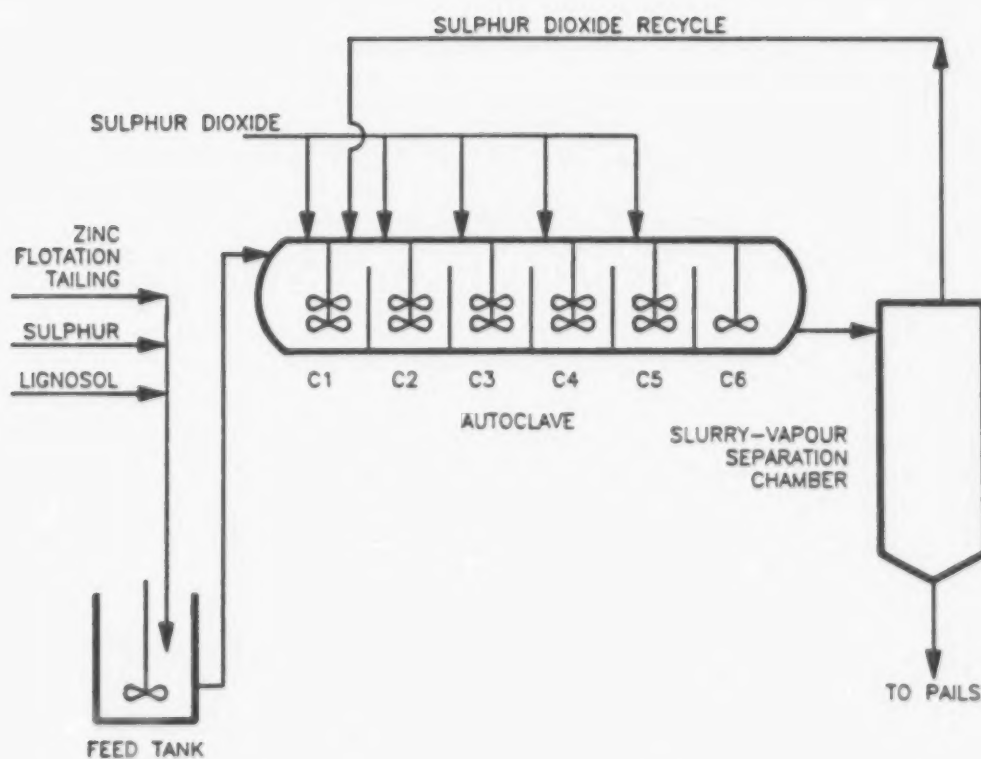


Figure 6.5 Continuous Reducing Leach Equipment Flowsheet

7.0 HOT ACID LEACH

Zinc oxide fume leach residue (oxide residue) was leached in spent electrolyte in the hot acid leach step. The process objectives were:

- to partially extract zinc, iron and copper from the oxides in the residue;
- to obtain high extraction of copper from the sulphides in the residue; and
- to minimize the formation of plumbojarosite by maintaining a high solution acid tenor.

Five batch tests were performed to establish the starting conditions for the continuous test. The continuous hot acid leach circuit was operated for 55 h, with 100% on stream time. 522 L of electrolyte and 276 L of slurry, containing 195 kg of solids, were processed through the continuous circuit.

7.1 FEED MATERIALS

Two samples of oxide residue from Flin Flon were treated in this phase of the project. The first sample, which was used for the first few batch tests, was of ponded residue and was received during the Phase Ib testwork (December 8, 1987). The second sample, representing current production, was received on March 15, 1989. Chemical analyses and moisture contents for the two samples are summarized below; the analysis for the March 15 sample is a mean of eight analyses of samples collected during the continuous leach test. Detailed analyses are provided in Section 5.

Sample	Analysis, % (dry basis)								Moisture %
	As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn	
Dec. 5, 1987	3.1	0.2	6.2	1.4	10.9	15.9	4.0	8.9	41.2
Mar. 15, 1989	1.3	0.1	6.4	1.6	14.5	13.7	4.7	11.5	46.0

The leach feed solutions used in the batch tests were synthetic spent electrolyte, or Cominco spent electrolyte fortified with sulphuric acid.

For the continuous test, purified neutral leach solution from Flin Flon was diluted to about 60 g/L Zn with demineralized water to represent the zinc tenor of HBM&S spent electrolyte. A portion of this solution was used to repulp the oxide residue to provide an appropriate pulp density to allow the slurry to be fed to the reaction vessel with a peristaltic pump. Since this slurry was to sit in pails for several days, the sulphuric acid that would be present in the solution if spent electrolyte were used to repulp the oxide residue was added instead to the remainder of the diluted, purified neutral leach solution to prevent

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premature leaching. Acid was added at such a concentration (152 g/L H_2SO_4) that the combination of this solution and the solution added to repulp the solids represented a net addition of spent electrolyte of approximate composition 60 g/L Zn and 135 g/L H_2SO_4 (as used by HBM&S at Flin Flon). Actual analyses for the feed solution and calculated analyses for the net solution added based on the slurry and solution feed rates are given below. Detailed analyses of the purified neutral leach solution are given in Section 5.

Sample	Analysis, g/L						
	As	Cd	Ca	Cu	Fe	H_2SO_4	Zn
Electrolyte Feed	<0.02	<0.005	0.15	<0.005	<0.005	152	64.4
Electrolyte + Repulp *							
Period 1	<0.02	<0.005	0.15	<0.005	<0.005	135	64.4
Period 2	<0.02	<0.005	0.15	<0.005	<0.005	138	64.4

* Calculated

7.2 BATCH TESTS

Five hot acid leach tests were carried out batchwise at 90°C in an open stainless steel beaker. Results are reported in Tables 7.1 to 7.5. The feed solutions ranged in acid strength from 182 to 286 g/L H_2SO_4 . The acid addition rate ranged from 170 to 326 kg/t of oxide residue. With the exception of the first test, which had the lowest acid addition, between 4 and 6 hours retention time was required to reduce the acid in solution to the target of about 50 g/L H_2SO_4 . The results for 6 h retention are summarized below.

Test	Feed Residue	Feed		Discharge H_2SO_4 g/L	H_2SO_4 Consump kg/t	Extraction		
		Pulp Density g/L *	H_2SO_4 Addition kg/t			Cu %	Fe %	Zn %
1	Old	611	170	44	98	58	16	37
2	Old	692	213	56	132	63	26	42
3	Old	694	204	50	132	50	26	44
4	New	446	326	54	206	76	35	29
5	New	424	323	52	202	74	34	28

* g solids per L solution

Insufficient acid was added in the first test and the iron extraction was only 16%. More acid was used in the second test and the iron extraction increased to 26%. Acid consumption was 132 kg/t solids, and the terminal acid concentration was 56 g/L H_2SO_4 . The third test was carried out on a larger scale than the first two tests to produce sufficient

feed slurry for neutralization-reduction tests. Less acid was added so that a terminal acidity of 50 g/L H_2SO_4 was obtained.

Two additional batch tests were carried out when the second sample of oxide residue was received. The acid consumption for this material was about 200 kg/t (Tests 4 and 5), which was significantly higher than for the previous sample. Iron and copper extractions were correspondingly higher for the new sample, (35% and 75%, respectively). Zinc extraction (28%) was lower for the new sample, suggesting that a larger percentage of the zinc in this sample was present as sphalerite or relatively inert phases such as zinc aluminate or silicate. The extractions resulting from treatment of the second sample and the product compositions are summarized below (Test 5).

Component	As	Cd	Cu	Fe^{2+}	Fe	Pb	H_2SO_4	Zn
Solution, g/L	5.4	0.2	6.1	5.8	25.0	-	51.5	61.6
Residue, %	0.4	0.1	0.5	-	11.8	19.6	-	10.8
Extraction, %	69	26	74	-	34	-	-	28

The concentration of zinc in the discharge solution was 61.6 g/L, compared with 56.5 g/L Zn in the feed solution. The small net zinc pickup was a result of dilution with the large quantity of water contained in the oxide residue feed. The discharge solution contained 25 g/L Fe, of which nearly 20 g/L was in the ferric state. Reduction of this iron and neutralization of the acid in the solution with zinc concentrate would allow for a significant increase in the zinc concentration of the solution in the next step of the process.

Solution compositions and extraction profiles for Test 5 are given in Figures 7.1 and 7.2 respectively. The bulk of the reaction is completed within the first 30 minutes, but acid concentrations are still decreasing and iron and zinc extractions are increasing after 6 hours.

7.3 CONTINUOUS HOT ACID LEACH TEST

7.3.1 Circuit Operation

A detailed description of the equipment and procedures for the hot acid leach circuit is provided in Section 6. The equipment flowsheet is provided in Figure 7.3. Continuous hot acid leaching was conducted in a train of four agitated stainless steel tanks arranged in cascade, each with an operating volume of 30 L. Oxide residue was pumped as a 45% solids slurry to the first tank of the train (HAL 1). Spent electrolyte was pumped separately to the same tank. An equal quantity of water was pumped to each tank to make up for evaporative losses, limiting the net loss to about 5%. Heat was applied by means of steam coils in the first and third tanks (HAL 1 and HAL 3), and by electric heating tapes on the second and fourth tanks (HAL 2 and HAL 4). Overflow slurry from HAL 4 was pumped to the neutralization-reduction circuit feed tank.

At startup, HAL 1 was filled with 16.2 kg of oxide residue slurry and 18.0 L of spent electrolyte. The mixture was agitated at 90°C for 1 h before starting to feed continuously. Tanks HAL 2, HAL 3 AND HAL 4 were allowed to fill with overflow slurry from the previous tanks in the train.

7.3.2 Sampling Procedures

For control purposes, a sample was taken every hour from the last tank, the filtrates were titrated for Fe^{2+} and H_2SO_4 and analysed by ICP for total iron and copper. Profile samples were taken every six hours from the four reaction tanks. The profile solutions were titrated for Fe^{2+} and H_2SO_4 and were subsequently analyzed for As, Cd, Ca, Cu, Fe and Zn. Profile solids were analyzed for As, Cd, Ca, Cu, Fe, Pb, SiO_2 and Zn.

7.3.3 Extraction Calculations

Copper, iron and zinc extractions in the hot acid leach were calculated using a lead tie, as shown below for zinc.

$$\% \text{ Zn Extraction} = \left[1 - \frac{\% \text{ Zn Residue}}{\% \text{ Zn Feed}} \times \frac{\% \text{ Pb Feed}}{\% \text{ Pb Residue}} \right] \times 100$$

Zinc extraction values can be adjusted to account for zinc contained in the solution component of the oxide residue filter cake feed (see Section 7.3.7).

7.3.4 History of Events

A chronological summary of parameter changes and activities during the continuous hot acid leach test is provided in the following section. Operations were uneventful with 100% on stream time.

Run Time h	Events and Changes
-0.5	Added 16.2 kg feed slurry and 18.0 L feed solution to tank HAL 1. Started agitation and steam.
0	Contents of HAL 1 at temperature (90°C).
1.0	Started continuous feed of slurry and solution to HAL 1 at target values: Slurry: 180 g/min Solution: 200 mL/min Water: 10 mL/min to each operating tank.
3.0	Decreased water flow to 5 mL/min per tank to better match target of 5% evaporation across the circuit.

Run Time h	Events and Changes															
5.8	All tanks full; started feeding Neutralization-Reduction circuit from HAL 4.															
10.5	Shut off water feed.															
27.5	Started collecting HAL 4 discharge in pails to decrease surge between Hot Acid Leach and Neutralization-Reduction.															
28.0	Decreased feed rates. New rates: Slurry: 79 g/min Solution: 110 mL/min															
40.0	Started water to tanks HAL 1 to HAL 4, total flow 15 mL/min (3.75 mL/min per tank).															
46.0	Decreased water flow to 10 mL/min.															
55.0	Shutdown. Shut off feeds and steam, collected contents of each tank separately.															
	<table><tr><td>Tank</td><td>Slurry Weight, kg</td><td>P.D., g/L</td></tr><tr><td>HAL 1</td><td>36.9</td><td>1404</td></tr><tr><td>HAL 2</td><td>36.6</td><td>1394</td></tr><tr><td>HAL 3</td><td>36.5</td><td>1387</td></tr><tr><td>HAL 4</td><td>38.8</td><td>1422</td></tr></table>	Tank	Slurry Weight, kg	P.D., g/L	HAL 1	36.9	1404	HAL 2	36.6	1394	HAL 3	36.5	1387	HAL 4	38.8	1422
Tank	Slurry Weight, kg	P.D., g/L														
HAL 1	36.9	1404														
HAL 2	36.6	1394														
HAL 3	36.5	1387														
HAL 4	38.8	1422														
	A total of 164 g of oversize material (>1.0 mm) was collected on the slurry feed tank screen.															

7.3.5 Operating Parameters

The hot acid leach operating parameters are given in Tables 7.6 and 7.7. A summary table is provided below.

Period	Run Time h	Temperature °C	Retention Time h	H ₂ SO ₄ Addition kg/t
1	0 to 28	90	6.5	377
2	28 to 55	90	12.7	478

In Period 1, the oxide residue slurry was fed to HAL 1 at a rate of 114 mL/min. Spent electrolyte was added at 200 mL/min. Additional feed details are as follows.

Period	Feed Slurry			Feed Solution mL/min	Feed + Repulp Solution mL/min	Electrolyte Rate m ³ /t solids
	Solids g/min	Entrained Solution mL/min	Repulp Solution mL/min			
1	80.5	68.6	25.1	200	225.1	2.80
2	35.3	30.1	11.0	111	122.1	3.46

Water was added initially in Period 1 to compensate for water lost by evaporation, but the evaporation rate was lower than anticipated and the water was shut off after 10 h. In this period the average water addition was 6.7 mL/min. The temperature in all four tanks was maintained at about 90°C.

In Period 2 the feed rates were decreased to increase the retention time in the circuit. The flow rate of the spent electrolyte was increased relative to the oxide residue to allow for increased consumption of acid while maintaining about 50 g/L H₂SO₄ in the discharge solution.

7.3.6 Acid Consumption

The acid concentration in the hot acid leach solution was monitored hourly, Tables 7.8 and 7.9, and profiles across the circuit were obtained every six hours, Tables 7.10 and 7.11. The results are plotted in Figures 7.4 and 7.5 and are summarized below.

Period	Retention Time h	H ₂ SO ₄ Addition kg/t	H ₂ SO ₄ , g/L				H ₂ SO ₄ Consumption kg/t
			HAL 1	HAL 2	HAL 3	HAL 4	
1	6.5	377	62.7	58.3	56.6	53.4	190
2	12.7	478	66.4	57.8	52.9	48.4	283

The acid consumption in Period 1, with a retention time of 6.5 h, was 190 kg/t of oxide residue, which is similar to that observed in the batch tests with 6 h retention (202 to 206 kg/t). The terminal acidity was 53.4 g/L H₂SO₄, very close to the target of 50 g/L.

Increasing the relative acid addition rate and the retention time in Period 2 had a significant impact on the acid consumption, which increased to 283 kg/t oxide residue. The average HAL 4 acid concentration in Period 2 was 48.4 g/L H₂SO₄. Although the acid concentration was lower in HAL 4 (12 h retention) than in HAL 3 (9 h retention), there was little difference in the metals extractions in these two tanks in Period 2 and thus there appears to be no advantage to increasing the retention time beyond about nine hours (see following section).

7.3.7 Metals Extractions

Profile solids compositions and extractions are given in Tables 7.12 to 7.14. Copper, iron and zinc extraction profiles are plotted in Figures 7.6 to 7.8 and terminal extractions are summarized below.

Period	Retention Time h	H ₂ SO ₄ Addition kg/t	Extraction, %				
			As	Cd	Cu	Fe	Zn
1	6.5	377	72	38	65	32	25
2	12.7	478	73	39	69	46	34

The conditions in Period 1 were very similar to those of the batch tests (See Section 7.2). Copper extraction during Period 1 was nearly 10% lower than in the batch tests (74% in Test 5), but the iron and zinc extractions were very similar. Increasing the retention time in Period 2 resulted in significant increases in the iron and zinc extractions, to 46% and 34% respectively.

About 70% of the arsenic and copper were leached in both periods, with most of the extraction taking place in the first tank. Iron and zinc leached more slowly, but leaching was essentially complete by the third tank during Period 2, representing 9 hours retention.

The head analysis used in the above extraction calculations was the average feed solids analysis determined during the continuous test. Analysis of the solution component of the feed slurry indicates, however, that a portion of the zinc in the feed was present in the solution component of the original feed cake, or was dissolved during repulping of the cake with zinc sulphate solution. The concentration of metals in the solution contained with the oxide residue feed, or the equivalent extracted during repulping is outlined below. The zinc content of the entrained solution represents 6.7% of the total zinc in the feed. Total zinc extracted into the hot acid leach discharge solution was 30% and 38% of the zinc in the oxide residue feed in Periods 1 and 2, respectively, compared with 25% and 34%, if the washed feed solids only are considered.

Feed Slurry Component	Analysis, g/L					
	As	Cd	Ca	Cu	Fe	Zn
Feed Slurry Solution	<0.02	0.05	0.51	0.07	0.01	24.3
Repulp Solution	<0.02	<0.01	0.15	<0.01	<0.01	64.4
Entrained Solution*	<0.02	0.07	0.64	0.10	0.10	9.63

* Calculated by difference.

7.4 CONCLUSIONS AND RECOMMENDATIONS

Copper, iron and zinc extractions in the hot acid leach step were about 70%, 45% and 35% respectively, with a retention time of 9 h at 85°C. The acid consumption in the continuous test was about 300 kg H₂SO₄ per tonne of oxide residue treated.

Acid may be conveniently provided to the hot acid leach by recycle of zinc spent electrolyte from the cellhouse. A terminal acidity of at least 50 g/L H₂SO₄ is desirable for rapid leaching, and to prevent reprecipitation of iron from solution.

Table 7.1 Hot Acid Leach: Batch Test 1

Charge	:	2.0 kg HBM&S oxide residue, 41.2% moisture (December 7, 1987 sample); 1.1 L fortified zinc spent electrolyte (52.9 g/L Zn; 182 g/L H ₂ SO ₄)
Solids Concentration	:	611 g/L solution
H ₂ SO ₄ Addition	:	170 kg/t oxide residue
Temperature	:	90°C
Equipment	:	Stainless steel beaker with mechanical agitation

	Retention Time, h						
	Head	0.25	0.5	1.0	2.0	4.0	6.0
Solutions, g/L							
As		6.2	6.4	5.5	5.4	5.3	4.6
Cd		0.55	0.56	0.49	0.50	0.51	0.49
Ca		0.98	0.87	0.80	0.77	0.72	0.69
Cu		4.94	5.38	4.89	5.13	5.27	5.05
Fe ²⁺		2.2	2.4	2.4	2.6	2.7	3.0
Fe		9.87	10.5	9.31	9.77	10.3	9.88
H ₂ SO ₄	104	60	60	54	53	51	44
Zn	30.2	51.3	53.6	47.7	48.8	50.0	48.3
Solids, %							
As	3.06						2.54
Cd	0.16						0.08
Ca	6.18						6.84
Cu	1.44						0.71
Fe	10.9						10.8
Pb	15.9						18.8
SiO ₂	3.96						4.71
S(SO ₄)	7.80						7.17
S	8.54						9.54
Zn	8.94						6.63
Extraction*, %							
Cu							58
Fe							16
Zn							37

* Based on lead tie.

Table 7.2 Hot Acid Leach: Batch Test 2

Charge	:	2.0 kg HBM&S oxide residue, 41.2% moisture (December 7, 1987 sample); 0.875 L of fortified zinc spent electrolyte (49.9 g/L Zn; 286 g/L H ₂ SO ₄)
Solids Concentration	:	692 g/L solution
H ₂ SO ₄ Addition	:	213 kg/t oxide residue
Temperature	:	90°C
Equipment	:	Stainless steel beaker with mechanical agitation

		Retention Time, h						
		Head	0.25	0.5	1.0	2.0	4.0	6.0
Solutions, g/L								
As			7.5	7.4	7.0	6.7	6.5	6.6
Cd			0.59	0.57	0.57	0.55	0.55	0.57
Ca			0.82	0.97	0.89	0.84	0.85	0.80
Cu			5.66	5.97	6.13	5.95	5.96	6.24
Fe ²⁺			2.2	2.8	3.3	3.3	3.4	3.4
Fe			13.8	14.7	15.7	16.0	16.8	18.3
H ₂ SO ₄	147		79	78	68	61	56	56
Zn	25.7		47.7	48.5	47.9	46.3	46.4	49.0
Solids, %								
As	3.06							2.42
Cd	0.16							0.08
Ca	6.18							6.88
Cu	1.44							0.66
Fe	10.9							9.86
Pb	15.9							19.4
SiO ₂	3.96							5.31
S(SO ₄)	7.80							8.10
S	8.54							9.41
Zn	8.94							6.34
Extraction*, %								
Cu								63
Fe								26
Zn								42

* Based on lead tie.

Table 7.3 Hot Acid Leach: Batch Test 3

Charge	:	5.65 kg HBM&S oxide residue, 41.2% moisture (December 7, 1987 sample); 2.46 L fortified zinc spent electrolyte (50.2 g/L Zn; 276 g/L H ₂ SO ₄)
Solids Concentration	:	694 g/L solution
H ₂ SO ₄ Addition	:	204 kg/t oxide residue
Temperature	:	90°C
Equipment	:	Stainless steel beaker with mechanical agitation

	Retention Time, h						
	Head	0.25	0.5	1.0	2.0	4.0	6.0
Solutions, g/L							
As		4.6	4.5	4.2	4.1	3.9	3.9
Cd		0.50	0.51	0.49	0.49	0.50	0.50
Ca		1.04	0.90	0.90	0.82	0.74	0.71
Cu		4.23	4.58	4.74	4.94	4.99	5.10
Fe ²⁺		2.4	3.0	3.6	4.2	4.0	4.4
Fe		11.5	12.7	13.7	15.8	17.7	19.1
H ₂ SO ₄	142	84	80	70	62	52	50
Zn	25.8	49.7	50.2	49.3	50.9	51.2	52.7
Solids, %							
As	3.06						2.61
Cd	0.16						0.09
Ca	6.18						6.37
Cu	1.44						0.84
Fe	10.9						9.35
Pb	15.9						18.3
SiO ₂	3.96						4.35
S(SO ₄)	7.80						7.96
S	8.54						9.61
Zn	8.94						5.78
Extraction*, %							
Cu							50
Fe							26
Zn							44

* Based on lead tie.

Table 7.4 Hot Acid Leach: Batch Test 4

Charge	:	3.0 kg HBM&S oxide residue, 46.0% moisture (March 15, 1989 sample); 2.25 L fortified zinc spent electrolyte (61.1 g/L Zn; 235 g/L H ₂ SO ₄)
Solids Concentration	:	446 g/L solution
H ₂ SO ₄ Addition	:	326 kg/t oxide residue
Temperature	:	90°C
Equipment	:	Stainless steel beaker with mechanical agitation

	Retention Time, h					
	Head	0.5	1.0	2.0	4.0	6.0
Solutions, g/L						
As		6.34	5.69	5.15	5.73	6.27
Cd		0.23	0.22	0.20	0.24	0.28
Ca		0.57	0.50	0.53	0.54	0.68
Cu		6.15	5.96	5.64	6.40	7.02
Fe ²⁺		2.4	3.0	3.5	4.8	5.4
Fe		21.9	21.7	21.5	25.7	29.5
H ₂ SO ₄	146	74.3	65.7	60.8	58.7	53.8
Zn	37.9	63.1	58.6	54.9	63.3	70.2
Solids, %						
As	1.16	0.33	0.34	0.35	0.37	0.39
Cd	0.11	0.10	0.10	0.10	0.10	0.10
Ca	6.55	7.54	7.75	7.63	7.86	7.88
Cu	1.65	0.64	0.56	0.52	0.50	0.49
Fe	14.5	12.7	12.2	12.0	11.7	11.5
Pb	15.9	18.8	18.9	19.3	19.1	19.4
SiO ₂	4.79	4.44	4.50	4.86	5.60	5.92
Zn	12.1	11.6	11.1	10.9	10.7	10.5
Extraction*, %						
Cu		67	71	74	75	76
Fe		26	29	32	33	35
Zn		19	23	26	26	29

* Based on lead tie.

Table 7.5 Hot Acid Leach: Batch Test 5

Charge	:	4.0 kg HBM&S oxide residue, 46.0% moisture (March 15, 1989 sample); 3.25 L fortified zinc spent electrolyte (56.5 g/L Zn; 215 g/L H ₂ SO ₄)
Solids Concentration	:	424 g/L solution
H ₂ SO ₄ Addition	:	323 kg/t oxide residue
Temperature	:	90°C
Equipment	:	Stainless steel beaker with mechanical agitation

	Retention Time, h					
	Head	0.5	1.0	2.0	4.0	6.0
Solutions, g/L						
As		5.56	5.54	5.13	5.35	5.41
Cd		0.21	0.22	0.22	0.24	0.24
Ca		0.55	0.48	0.49	0.65	0.65
Cu		5.48	5.87	5.56	5.97	6.10
Fe ²⁺		2.5	3.4	4.1	5.1	5.8
Fe		19.1	20.7	20.5	23.2	25.0
H ₂ SO ₄	137	69	66.5	61	57	51.5
Zn	36.1	56.4	58.5	55.3	59.7	61.6
Solids, %						
As	1.16	0.38	0.57	0.50	0.54	0.45
Cd	0.11	0.10	0.10	0.10	0.10	0.10
Ca	6.55	7.45	7.65	7.34	7.40	7.33
Cu	1.65	0.63	0.57	0.52	0.52	0.52
Fe	14.5	12.6	12.4	11.8	11.9	11.8
Pb	15.9	18.7	19.4	18.5	19.2	19.6
SiO ₂	4.79	4.61	5.06	5.16	5.88	6.34
Zn	12.1	11.6	11.3	10.8	10.8	10.8
Extraction*, %						
Cu		68	72	73	74	74
Fe		26	30	30	32	34
Zn		18	23	23	26	28

* Based on lead tie.

Table 7.6 Hot Acid Leach Operating Parameters (Period 1)

Run Time h	Slurry Feed mL/min	Sol'n Feed mL/min	Water mL/min	Temperature				Discharge	
				HAL 1 °C	HAL 2 °C	HAL 3 °C	HAL 4 °C	Rate mL/min	P.D. g/L
1	115	200	10	90	—	—	—	—	—
2	115	202	20	88	78	—	—	—	—
3	115	200	20	88	83	—	—	—	—
4	121	202	15	88	87	91	—	—	—
5	114	200	22	88	91	91	88	—	—
6	116	200	21	88	88	90	86	320	1359
7	114	200	21	88	90	90	89	320	1351
8	114	200	21	89	90	91	89	322	1358
9	116	200	20	89	90	90	90	316	1361
10	114	200	20	89	90	90	90	322	1365
11	114	200	—	89	90	91	90	290	1362
12	114	205	—	88	90	91	92	322	1362
13	113	198	—	88	91	91	92	296	1365
14	115	199	—	88	92	91	91	302	1366
15	114	198	—	89	92	91	91	300	1374
16	115	198	—	88	92	92	92	266	1366
17	115	198	—	88	92	92	92	288	1380
18	113	197	—	88	92	92	92	298	1386
19	113	198	—	89	92	92	92	262	1382
20	113	197	—	88	92	92	92	290	1387
21	115	198	—	88	92	91	92	282	1398
22	113	201	—	88	92	91	92	272	1373
23	112	202	—	88	92	91	91	284	1380
24	113	202	—	89	92	91	91	284	1388
25	114	200	—	89	92	91	91	290	1384
26	114	200	—	89	92	92	91	290	1389
27	114	202	—	89	91	92	91	304	1392
28	114	200	—	89	92	92	91	305	1390
Avg	114	200	6.7	88	90	91	91	297	1375

Table 7.7 Hot Acid Leach Operating Parameters (Period 2)

Run Time h	Slurry Feed mL/min	Sol'n Feed mL/min	Water mL/min	Temperature				Discharge	
				HAL 1 °C	HAL 2 °C	HAL 3 °C	HAL 4 °C	Rate mL/min	P.D. g/L
29	50	111	—	89	91	91	92	135	1390
30	51	109	—	89	91	92	92	155	1394
31	50	110	—	89	91	91	91	138	1401
32	51	110	—	89	91	91	91	133	1405
33	51	110	—	89	90	90	90	136	1402
34	50	110	—	89	90	91	89	144	1401
35	51	110	—	89	90	90	89	121	1408
36	51	114	—	89	89	90	90	131	1390
37	51	113	—	88	90	91	91	145	1385
38	52	114	—	88	90	90	92	150	1385
39	53	113	—	88	89	90	92	152	1392
40	50	112	—	88	89	90	91	130	1404
41	49	110	—	88	90	90	91	137	1400
42	50	112	16	88	90	90	90	166	1387
43	51	111	15	89	90	90	90	148	1384
44	49	112	16	88	89	90	89	149	1383
45	50	111	16	88	89	90	89	143	1390
46	49	111	17	88	89	90	88	156	1382
47	49	111	10	88	90	90	88	153	1379
48	51	111	10	89	90	90	89	146	1379
49	51	112	10	89	90	90	89	145	1378
50	51	111	10	89	90	90	89	148	1382
51	51	111	10	87	90	90	89	152	1382
52	50	111	10	89	90	90	90	153	1378
53	50	111	10	89	89	90	89	145	1384
54	51	111	10	89	90	90	89	148	1383
55	50	111	10	89	90	90	89	153	1382
Avg	50	111	6.3	89	90	90	90	148	1389

Table 7.8 Hot Acid Leach Control Solution Analyses (Period 1)

Run Time h	Sample	Analysis, g/L			
		Cu	Fe ²⁺	Fe	H ₂ SO ₄
3	HAL 2	3.17	n.a.	11.0	58.7
4	HAL 3	3.28	2.0	11.9	61.8
5	HAL 3	3.28	2.1	11.9	58.8
6	HAL 4	3.29	2.2	12.3	59.0
7	HAL 4	3.23	2.1	12.5	54.0
8	HAL 4	3.23	2.1	12.6	53.5
9	HAL 4	3.11	2.2	12.1	55.8
10	HAL 4	3.00	2.2	12.1	53.0
11	HAL 4	2.81	2.1	11.1	48.5
12	HAL 4	2.86	2.0	11.3	49.0
13	HAL 4	3.05	2.2	12.2	55.2
14	HAL 4	2.78	2.0	11.3	50.2
15	HAL 4	3.12	2.2	12.8	55.5
16	HAL 4	3.11	2.3	13.2	54.7
17	HAL 4	3.19	2.3	13.3	54.0
18	HAL 4	2.97	2.2	12.4	50.5
19	HAL 4	3.25	2.3	13.6	54.8
20	HAL 4	3.26	2.4	13.6	55.2
21	HAL 4	3.22	2.3	13.5	53.0
22	HAL 4	3.28	2.4	14.1	53.2
23	HAL 4	3.27	2.3	13.7	53.2
24	HAL 4	3.33	2.4	14.0	54.5
25	HAL 4	3.27	2.4	13.8	54.5
26	HAL 4	2.97	2.4	12.6	48.8
27	HAL 4	3.16	2.2	13.6	50.0
28	HAL 4	3.25	2.3	13.9	52.5
Avg	HAL 4	3.14	2.2	12.7	53.9

n.a. = not analyzed

Table 7.9 Hot Acid Leach Control Solution Analyses (Period 2)

Run Time h	Sample	Analysis, g/L			
		Cu	Fe ²⁺	Fe	H ₂ SO ₄
29	HAL 4	3.24	2.4	14.2	53.0
30	HAL 4	2.98	2.3	13.2	49.0
31	HAL 4	3.16	2.3	14.4	47.8
32	HAL 4	3.56	2.5	16.6	53.3
33	HAL 4	3.47	2.5	16.6	51.3
34	HAL 4	3.29	2.5	16.3	49.3
35	HAL 4	3.27	2.5	16.8	47.3
36	HAL 4	3.43	2.5	18.2	49.0
37	HAL 4	3.42	2.5	18.6	48.0
38	HAL 4	4.00	2.5	22.3	48.2
39	HAL 4	3.81	2.5	21.8	48.2
40	HAL 4	3.40	2.5	20.3	48.7
41	HAL 4	3.31	2.5	19.8	48.5
42	HAL 4	3.64	2.4	22.1	49.0
43	HAL 4	3.76	2.5	23.1	48.2
44	HAL 4	3.42	2.4	21.3	48.2
45	HAL 4	3.07	2.4	19.4	47.5
46	HAL 4	3.13	2.4	20.2	48.0
47	HAL 4	2.94	2.3	18.7	45.8
48	HAL 4	3.16	2.5	20.2	46.9
49	HAL 4	3.04	2.4	19.4	47.8
50	HAL 4	2.99	2.3	19.1	49.3
51	HAL 4	3.07	2.4	19.7	48.0
52	HAL 4	2.94	2.3	18.6	47.3
53	HAL 4	n.a.	2.2	18.1	43.8
54	HAL 4	n.a.	2.4	19.3	48.8
55	HAL 4	2.97	2.3	19.1	48.6
Avg	HAL 4	3.30	2.4	18.8	48.5

n.a. = not analyzed

Table 7.10 Hot Acid Leach Profile Solution Analyses (Period 1)

Run Time h	Sample	Analysis, g/L							
		As	Cd	Ca	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Zn
4	Feed*	<0.02	0.05	0.49	0.07	n.a.	<0.01	n.a.	24.2
	HAL 1	2.76	0.12	0.78	2.87	1.8	9.85	62.3	56.6
	HAL 2	2.84	0.12	0.77	3.08	1.8	11.0	59.0	58.0
	HAL 3	2.99	0.12	0.73	3.28	2.0	11.9	61.8	60.3
10	Feed*	<0.02	0.06	0.52	0.08	n.a.	0.01	n.a.	24.5
	HAL 1	2.74	0.12	0.89	2.81	1.7	9.76	62.5	57.0
	HAL 2	2.68	0.12	0.76	2.91	2.0	10.5	59.0	56.6
	HAL 3	2.77	0.13	0.69	3.07	2.2	11.7	58.0	58.9
	HAL 4	2.69	0.12	0.68	3.00	2.2	12.1	53.0	57.2
16	Feed*	<0.02	0.06	0.51	0.08	n.a.	0.03	n.a.	24.1
	HAL 1	2.75	0.12	0.72	2.85	1.8	9.95	62.0	56.1
	HAL 2	2.69	0.12	0.64	2.95	2.1	11.0	58.8	57.1
	HAL 3	2.52	0.12	0.52	2.79	2.1	11.2	50.8	53.9
	HAL 4	2.78	0.13	0.60	3.11	2.3	13.2	54.7	60.4
22	Feed*	<0.02	0.05	0.49	0.07	n.a.	<0.01	n.a.	21.2
	HAL 1	2.78	0.12	0.78	2.86	1.7	10.0	63.5	57.2
	HAL 2	2.74	0.12	0.77	2.99	2.1	11.2	59.5	57.8
	HAL 3	2.92	0.13	0.74	3.22	2.3	13.0	56.7	61.7
	HAL 4	2.95	0.14	0.69	3.28	2.4	14.1	53.2	62.9
28	Feed*	<0.02	0.05	0.50	0.07	n.a.	<0.01	n.a.	24.1
	HAL 1	2.72	0.11	0.62	2.91	1.7	10.1	63.0	57.0
	HAL 2	2.60	0.11	0.54	2.92	1.9	10.8	55.3	55.6
	HAL 3	2.76	0.12	0.54	3.16	2.2	12.7	55.8	59.6
	HAL 4	2.81	0.13	0.52	3.25	2.3	13.9	52.5	60.8
Avg	Feed*	<0.02	0.05	0.51	0.07	n.a.	0.01	n.a.	24.3
	HAL 1	2.75	0.12	0.76	2.86	1.7	9.93	62.7	56.8
	HAL 2	2.71	0.12	0.70	2.97	2.0	10.9	58.3	57.0
	HAL 3	2.79	0.12	0.64	3.10	2.2	12.1	56.6	58.9
	HAL 4	2.81	0.13	0.62	3.16	2.3	13.3	53.4	60.3

* Solution contained with feed slurry. Mean feed values for entire run used for period average.

n.a. = not analyzed

Table 7.11 Hot Acid Leach Profile Solution Analyses (Period 2)

Run Time h	Sample	Analysis, g/L							
		As	Cd	Ca	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Zn
34	Feed*	<0.02	0.06	0.52	0.08	n.a.	<0.01	n.a.	24.2
	HAL 1	2.48	0.11	0.79	2.61	1.7	12.4	66.5	62.8
	HAL 2	2.64	0.12	0.80	2.90	2.0	14.6	59.3	64.2
	HAL 3	2.83	0.13	0.68	3.17	2.5	15.8	54.0	65.1
	HAL 4	2.94	0.14	0.62	3.29	2.5	16.3	49.3	64.7
40	Feed*	<0.02	0.06	0.52	0.08	n.a.	<0.01	n.a.	24.4
	HAL 1	2.44	0.11	0.79	2.58	1.7	12.2	66.7	61.9
	HAL 2	2.52	0.12	0.67	2.79	1.9	15.6	57.7	65.0
	HAL 3	3.13	0.16	0.81	3.56	2.2	21.5	53.0	80.3
	HAL 4	2.99	0.15	0.64	3.40	2.5	20.3	48.7	73.4
46	Feed*	<0.02	0.06	0.51	0.08	n.a.	0.03	n.a.	24.3
	HAL 1	2.33	0.10	0.77	2.49	1.6	11.8	66.2	59.8
	HAL 2	2.43	0.12	0.70	2.72	1.9	15.0	56.5	63.4
	HAL 3	2.80	0.14	0.77	3.19	2.2	19.6	52.0	73.1
	HAL 4	2.70	0.14	0.62	3.13	2.4	20.2	48.0	70.3
52	Feed*	<0.02	0.03	0.50	0.05	n.a.	<0.01	n.a.	24.6
	HAL 1	2.44	0.07	0.73	2.56	1.7	11.6	66.3	59.8
	HAL 2	2.44	0.08	0.66	2.67	2.0	14.2	57.8	61.7
	HAL 3	2.57	0.09	0.71	2.85	2.2	16.9	52.5	65.6
	HAL 4	2.60	0.09	0.63	2.94	2.3	18.6	47.3	67.4
55	HAL 4	2.64	0.12	0.69	2.97	2.3	19.1	48.6	67.4
Avg	Feed*	<0.02	0.05	0.51	0.07	n.a.	0.01	n.a.	24.3
	HAL 1	2.42	0.10	0.77	2.56	1.7	12.0	66.4	61.1
	HAL 2	2.51	0.11	0.71	2.77	2.0	14.8	57.8	63.6
	HAL 3	2.83	0.13	0.74	3.19	2.3	18.4	52.9	67.9
	HAL 4	2.77	0.13	0.64	3.15	2.4	18.9	48.4	68.6

* Solution contained with feed slurry. Mean feed values for entire run used for period average.

n.a. = not analyzed

Table 7.12 Hot Acid Leach Profile Solids Analyses (Period 1)

Run Time h	Sample	Analysis, %							
		As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn
4	Feed	1.27	0.11	6.26	1.60	14.2	13.4	4.65	11.3
	HAL 1	0.47	0.10	6.43	0.76	13.7	16.9	4.54	11.8
	HAL 2	0.41	0.10	6.66	0.65	13.1	17.2	4.54	11.3
	HAL 3	0.40	0.10	6.67	0.62	12.9	17.5	4.61	11.5
10	Feed	1.31	0.12	6.37	1.61	14.3	13.6	4.74	11.5
	HAL 1	0.46	0.10	6.83	0.78	13.4	16.5	4.37	11.5
	HAL 2	0.49	0.09	7.31	0.71	12.7	16.0	4.48	10.8
	HAL 3	0.46	0.09	7.11	0.70	12.6	16.4	4.52	10.8
	HAL 4	0.46	0.09	7.35	0.66	12.1	16.3	4.74	10.6
16	Feed	1.25	0.11	6.07	1.58	14.1	13.2	4.61	11.4
	HAL 1	0.42	0.09	6.75	0.76	13.2	16.2	4.20	11.2
	HAL 2	0.41	0.09	7.01	0.71	12.8	16.2	4.14	10.9
	HAL 3	0.43	0.09	6.93	0.70	12.6	16.7	4.31	10.7
	HAL 4	0.42	0.09	7.02	0.69	12.2	16.7	4.54	10.5
22	Feed	1.31	0.12	6.15	1.63	14.5	13.8	4.67	11.6
	HAL 1	0.44	0.09	6.92	0.76	13.2	16.2	4.18	11.3
	HAL 2	0.43	0.09	7.02	0.71	12.7	16.3	4.37	10.8
	HAL 3	0.46	0.09	7.40	0.68	12.2	16.6	4.44	10.4
	HAL 4	0.44	0.09	7.20	0.68	11.8	16.7	4.67	10.1
28	Feed	1.30	0.11	6.33	1.59	14.2	13.6	4.65	11.4
	HAL 1	0.47	0.10	6.96	0.73	13.2	16.7	4.24	11.3
	HAL 2	0.53	0.10	6.78	0.72	13.0	17.0	4.39	11.3
	HAL 3	0.50	0.10	7.29	0.69	12.3	16.9	4.54	10.7
	HAL 4	0.51	0.10	7.35	0.69	12.1	17.2	4.71	10.7
Avg	Feed*	1.33	0.12	6.35	1.61	14.5	13.7	4.71	11.5
	HAL 1	0.45	0.10	6.78	0.76	13.3	16.5	4.31	11.4
	HAL 2	0.45	0.09	6.96	0.70	12.9	16.5	4.38	11.0
	HAL 3	0.45	0.09	7.08	0.68	12.5	16.8	4.48	10.8
	HAL 4	0.45	0.09	7.23	0.68	12.0	16.7	4.66	10.5

* Mean feed values for entire run.

Table 7.13 Hot Acid Leach Profile Solids Analyses (Period 2)

Run Time h	Sample	Analysis, %							
		As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn
34	Feed	1.29	0.11	6.33	1.60	14.2	13.5	4.48	11.5
	HAL 1	0.41	0.10	6.61	0.73	12.4	18.0	4.67	11.2
	HAL 2	0.42	0.10	7.19	0.67	11.5	17.7	4.74	10.5
	HAL 3	0.43	0.09	7.28	0.66	11.4	17.5	4.74	10.3
	HAL 4	0.45	0.09	7.36	0.66	11.4	17.4	4.78	10.3
40	Feed	1.39	0.12	6.24	1.64	14.6	14.0	4.71	11.7
	HAL 1	0.52	0.10	6.69	0.76	12.6	17.7	4.82	11.2
	HAL 2	0.51	0.10	7.29	0.70	11.1	18.2	4.93	10.3
	HAL 3	0.49	0.10	7.10	0.69	10.8	19.2	5.55	10.1
	HAL 4	0.46	0.10	7.42	0.66	10.5	18.1	5.46	10.2
46	Feed	1.33	0.11	6.30	1.63	14.8	13.7	4.65	11.4
	HAL 1	0.46	0.10	6.08	0.80	13.4	18.2	4.86	11.4
	HAL 2	0.44	0.09	6.96	0.72	11.8	18.0	4.93	10.4
	HAL 3	0.42	0.10	6.55	0.71	11.2	18.9	5.66	10.1
	HAL 4	0.39	0.10	7.42	0.66	10.2	17.8	5.74	9.9
52	Feed	1.55	0.13	7.07	1.62	15.4	15.6	5.19	13.1
	HAL 1	0.59	0.11	7.49	0.78	13.6	20.0	5.14	12.7
	HAL 2	0.59	0.11	7.65	0.74	12.0	19.6	5.16	11.3
	HAL 3	0.58	0.11	7.12	0.75	11.5	20.9	5.68	10.9
	HAL 4	0.58	0.10	7.38	0.72	10.7	20.6	5.85	10.5
55	HAL 4	0.58	0.11	7.65	0.73	10.7	19.4	5.68	11.1
Avg	Feed*	1.33	0.12	6.35	1.61	14.5	13.7	4.71	11.5
	HAL 1	0.50	0.10	6.72	0.77	13.0	18.5	4.87	11.6
	HAL 2	0.46	0.10	7.27	0.71	11.6	18.4	4.94	10.6
	HAL 3	0.48	0.10	7.01	0.70	11.2	19.1	5.41	10.4
	HAL 4	0.49	0.10	7.45	0.69	10.7	18.7	5.50	10.4

* Mean feed values for entire run.

Table 7.14 Hot Acid Leach Profile Solids Extractions

Period 1					Period 2				
Run Time h	Sample	Extraction, % *			Run Time h	Sample	Extraction, % *		
		Cu	Fe	Zn			Cu	Fe	Zn
4	HAL 1	62	23	17	34	HAL 1	66	35	26
	HAL 2	68	28	22		HAL 2	68	39	30
	HAL 3	70	30	22		HAL 3	68	38	30
	HAL 4	n.s.	n.s.	n.s.		HAL 4	68	38	30
10	HAL 1	60	23	18	40	HAL 1	64	33	25
	HAL 2	63	25	20		HAL 2	68	42	33
	HAL 3	64	27	22		HAL 3	70	47	38
	HAL 4	66	30	23		HAL 4	69	45	33
16	HAL 1	60	23	18	46	HAL 1	63	30	26
	HAL 2	63	25	20		HAL 2	66	38	32
	HAL 3	65	29	24		HAL 3	68	44	37
	HAL 4	65	31	26		HAL 4	69	46	34
22	HAL 1	60	23	18	52	HAL 1	67	36	25
	HAL 2	63	26	22		HAL 2	68	42	32
	HAL 3	65	31	26		HAL 3	70	48	38
	HAL 4	66	33	28		HAL 4	70	51	40
28	HAL 1	63	25	20	55	HAL 1	n.s.	n.s.	n.s.
	HAL 2	64	28	21		HAL 2	n.s.	n.s.	n.s.
	HAL 3	66	31	25		HAL 3	n.s.	n.s.	n.s.
	HAL 4	66	34	26		HAL 4	68	48	32
Avg	HAL 1	61	24	18	Avg	HAL 1	65	34	25
	HAL 2	64	26	21		HAL 2	67	40	31
	HAL 3	66	30	23		HAL 3	69	45	35
	HAL 4	65	32	25		HAL 4	69	46	34

* Based on lead tie, and using mean analyses for feed solids.

n.s. = no sample

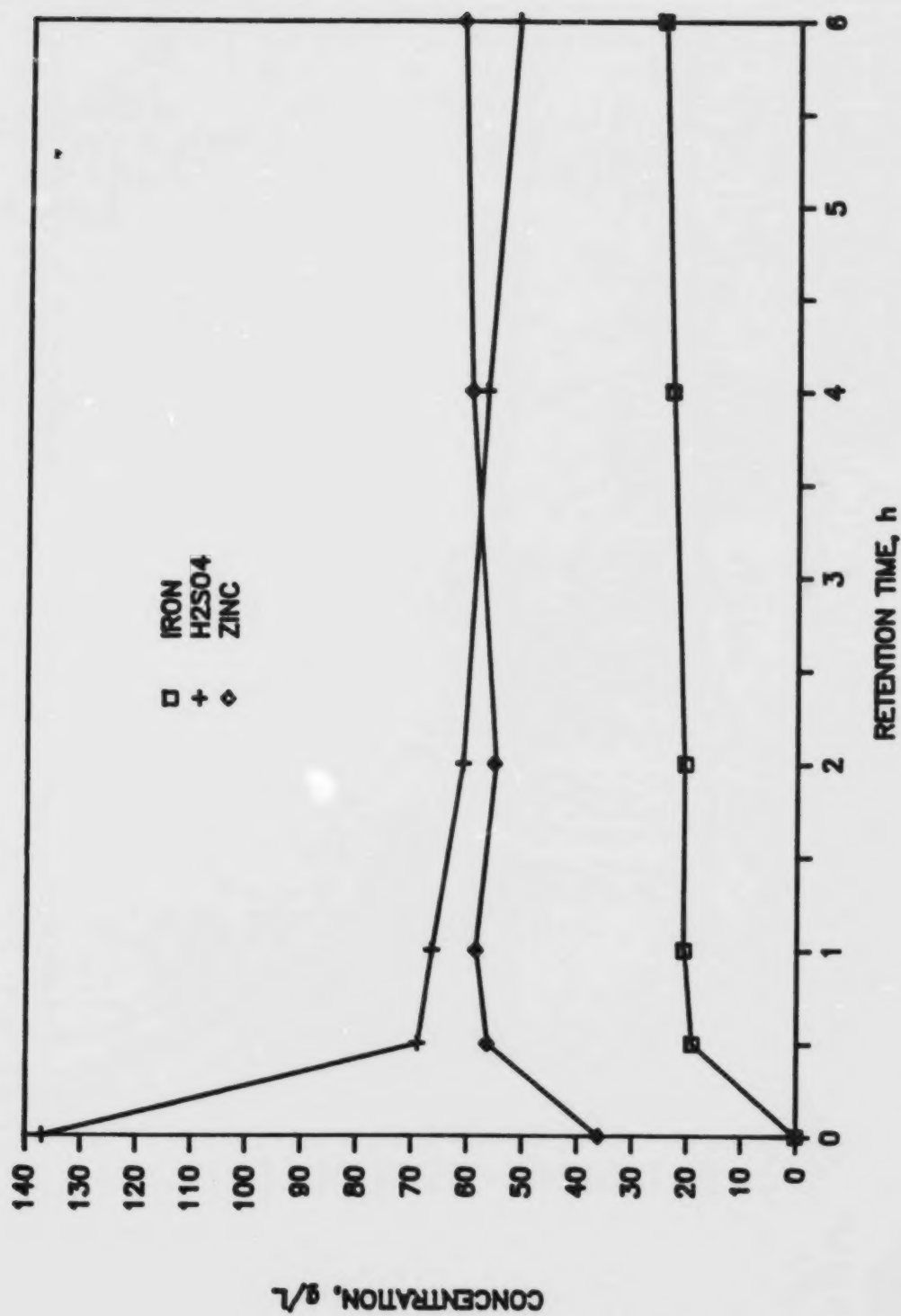


Figure 7.1 Solution Composition in Batch Test 5

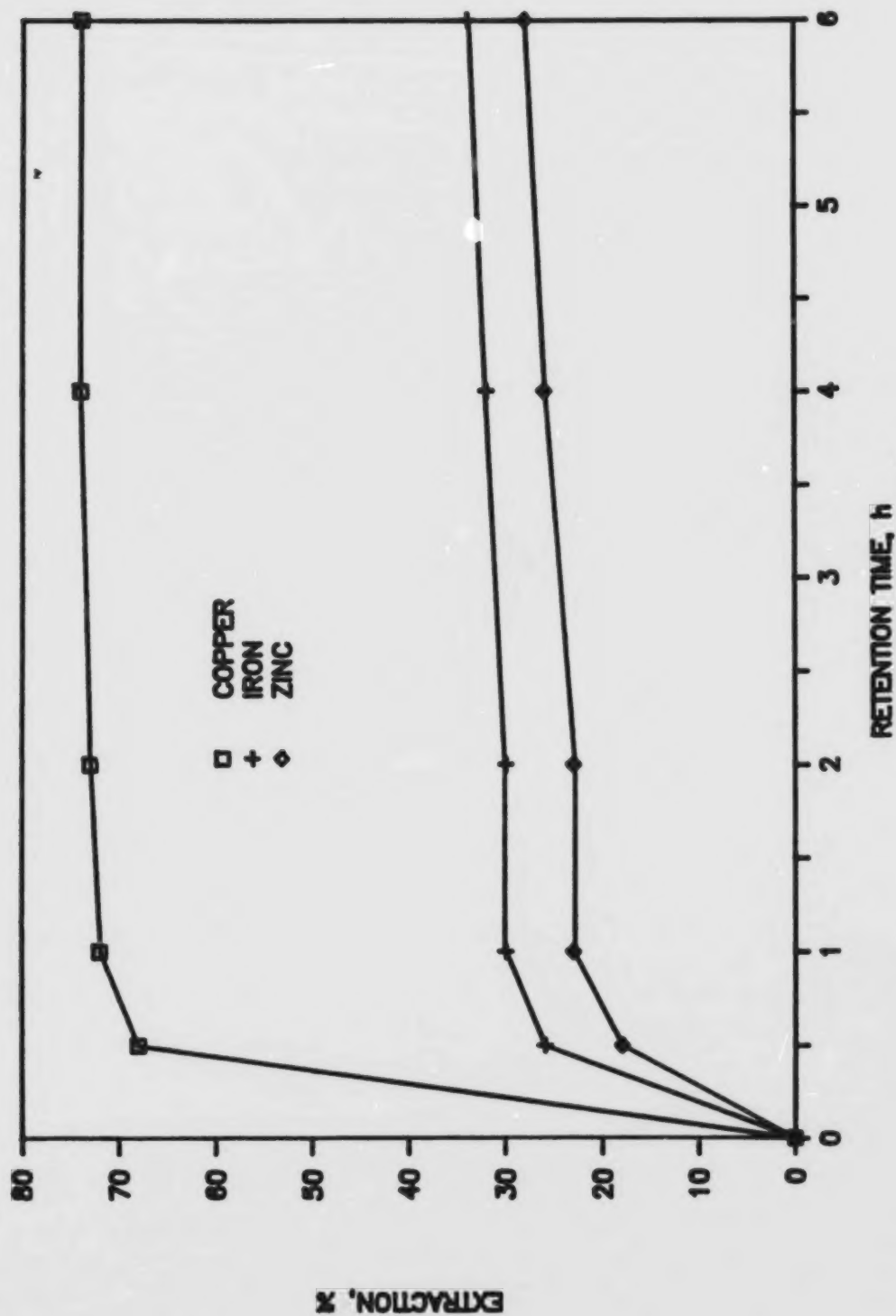


Figure 7.2 Metals Extractions in Batch Test 5

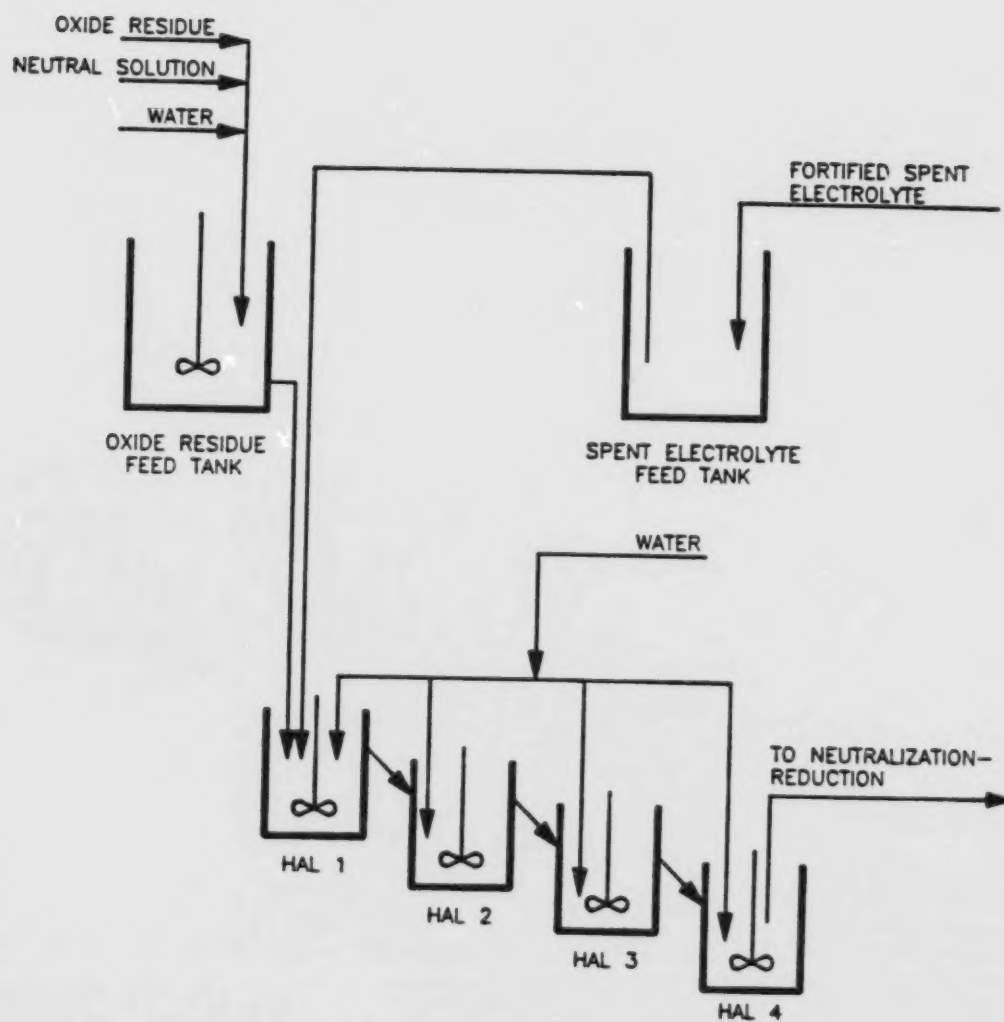


Figure 7.3 Continuous Hot Acid Leach Equipment Flowsheet

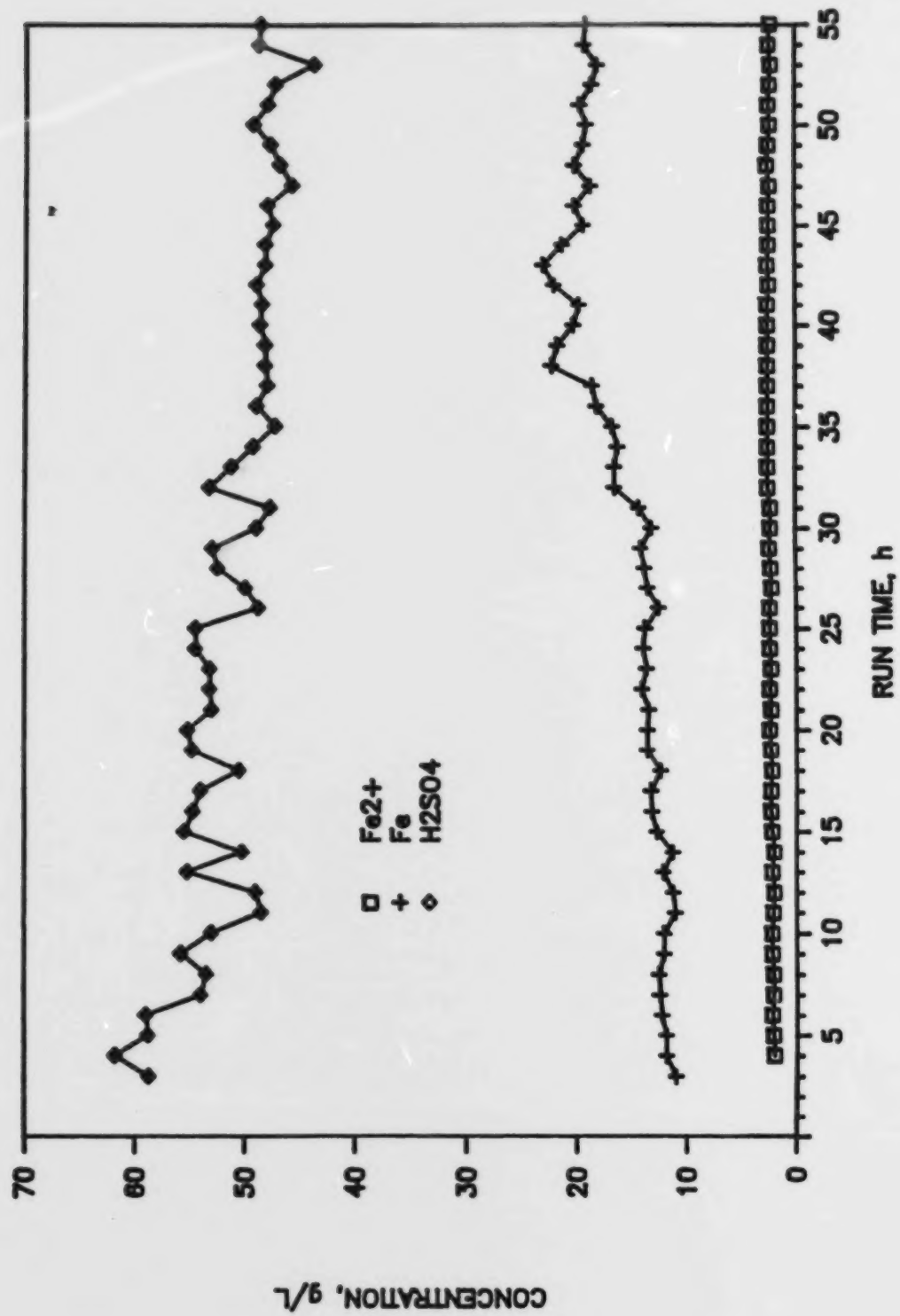


Figure 7.4 Solution Composition in HAL 4

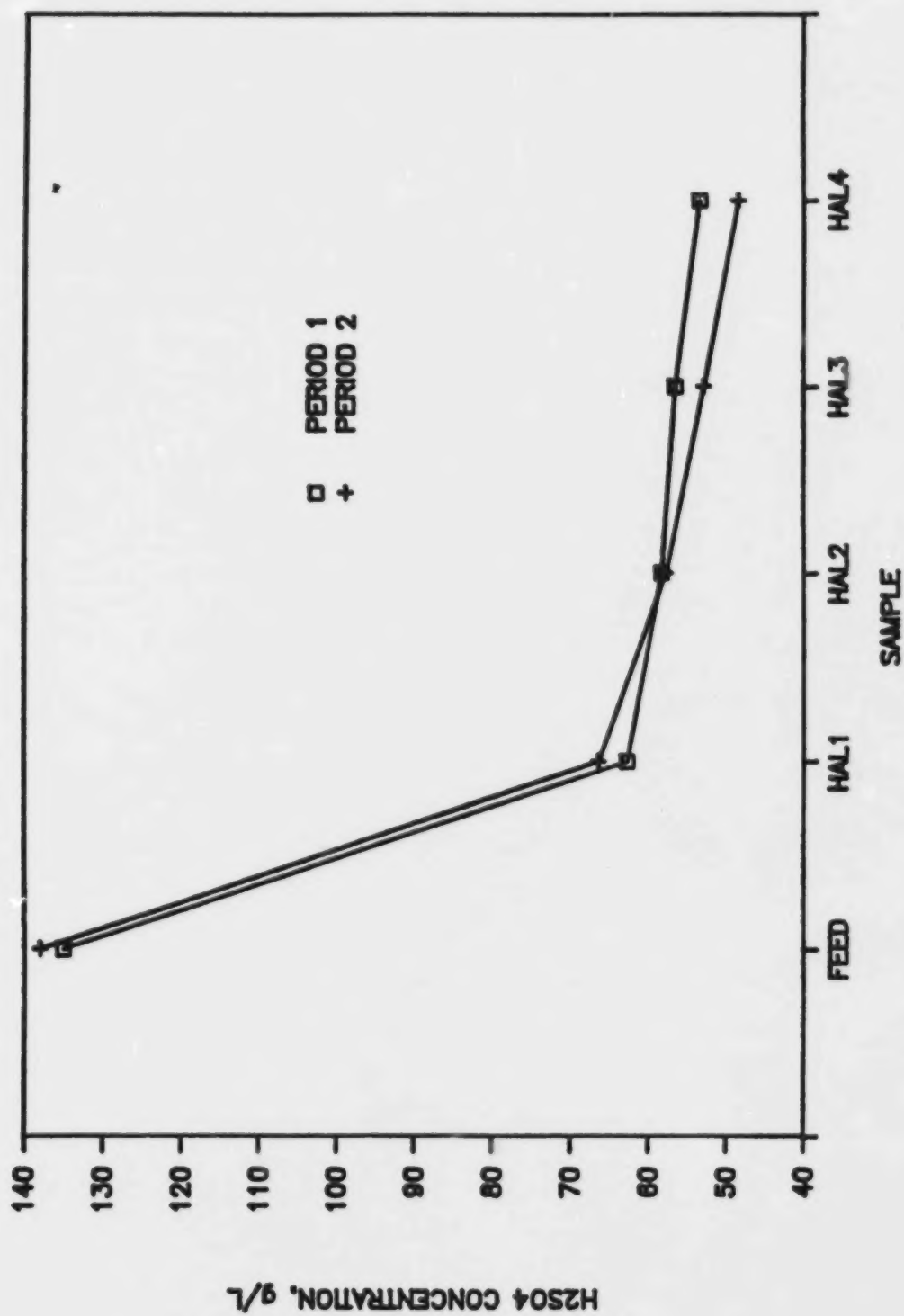


Figure 7.5 Continuous Hot Acid Leach Acid Profiles

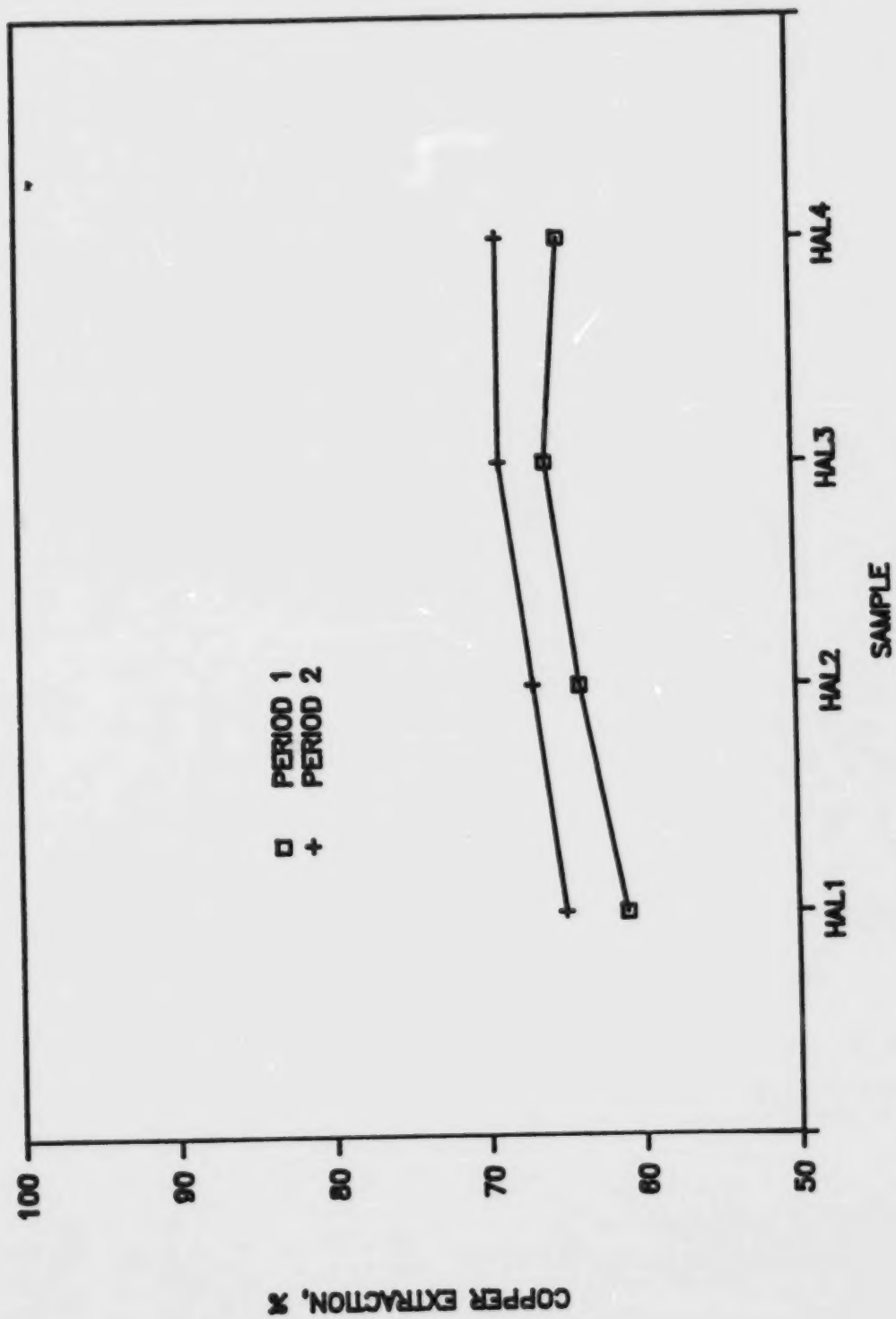


Figure 7.6 Continuous Hot Acid Leach Copper Extraction Profiles

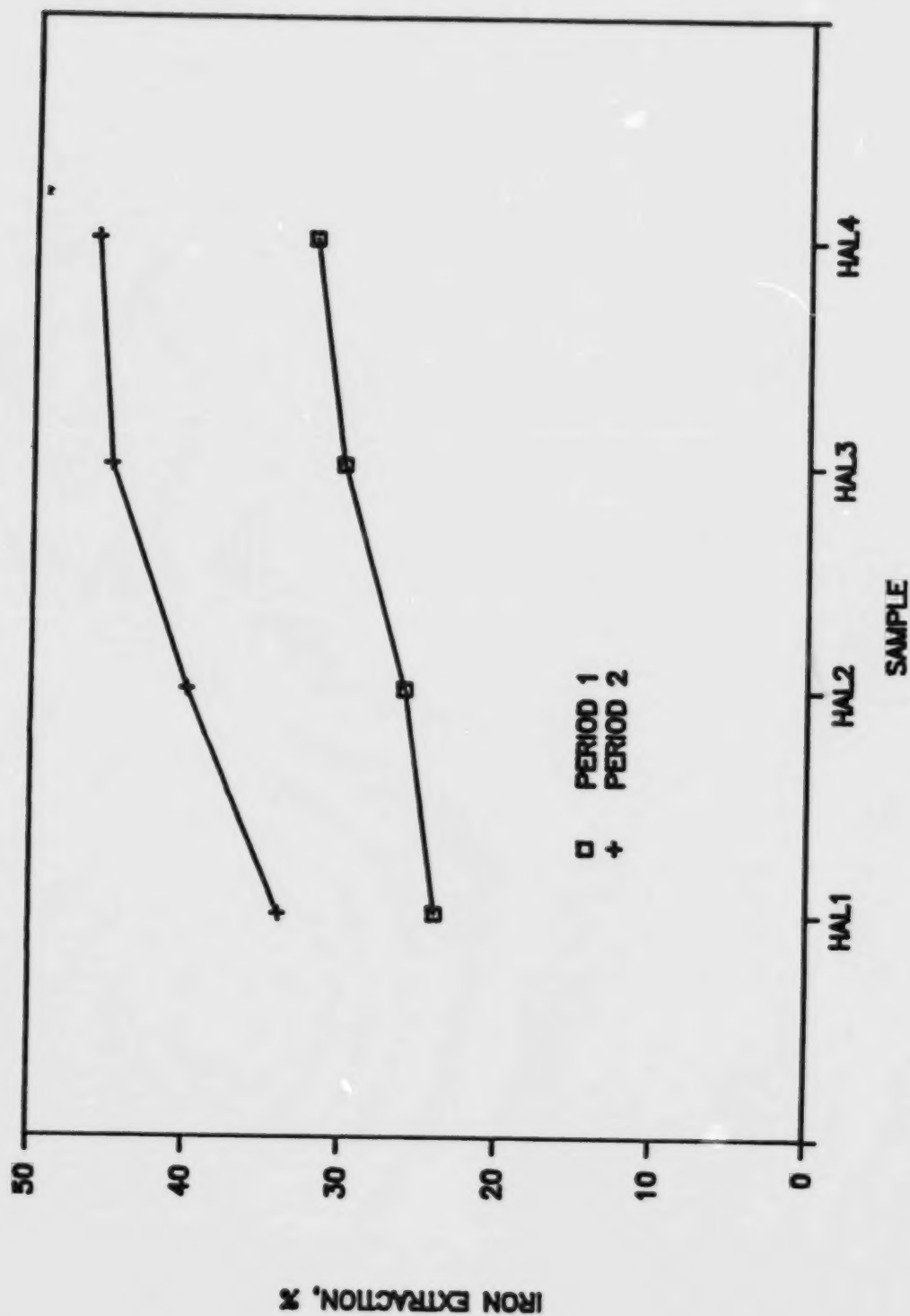


Figure 7.7 Continuous Hot Acid Leach Iron Extraction Profiles

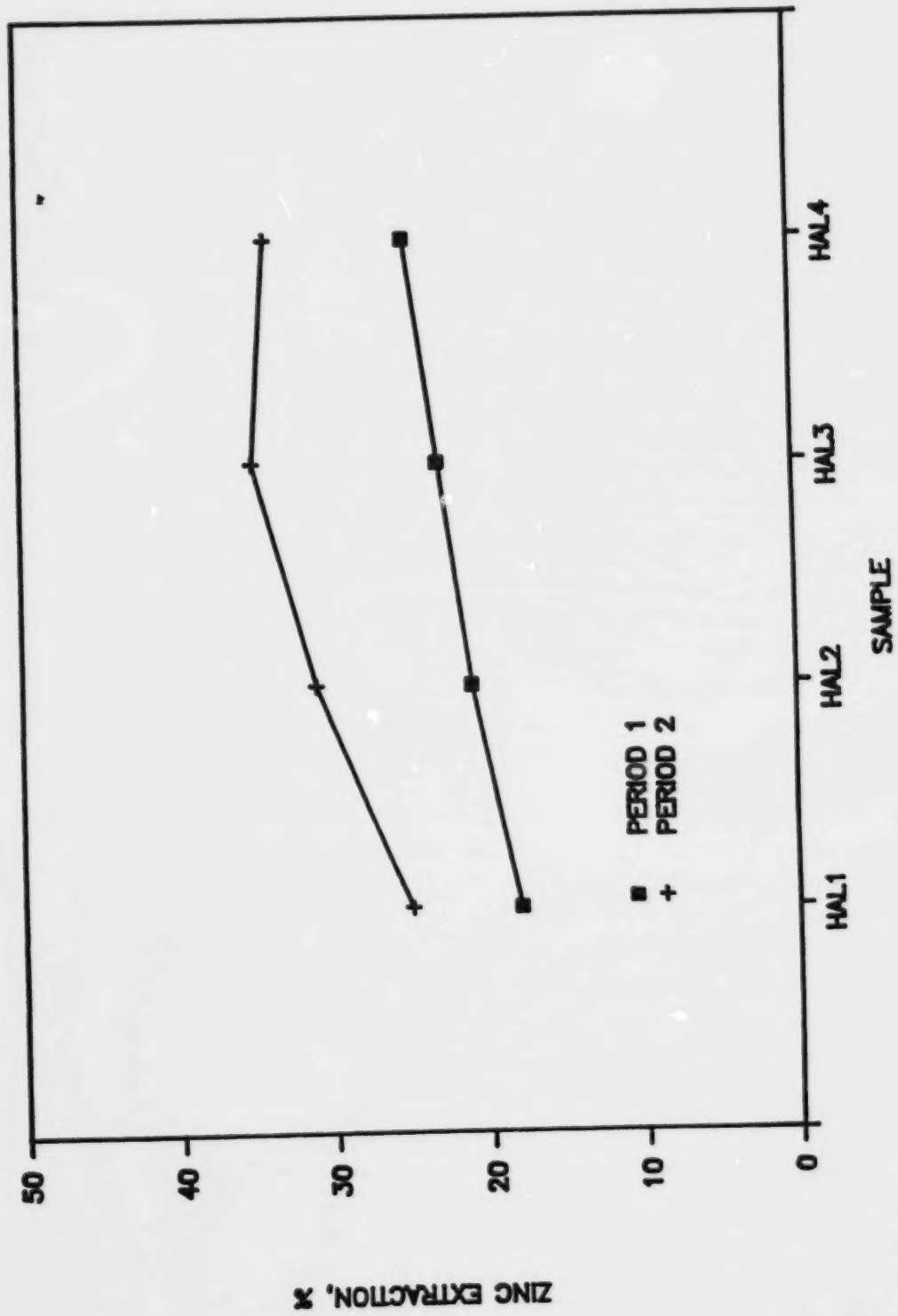


Figure 7.8 Continuous Hot Acid Leach Zinc Extraction Profiles

8.0 NEUTRALIZATION-REDUCTION

Discharge slurry from the hot acid leach was treated with zinc concentrate in the neutralization-reduction step. The process objectives were:

to decrease the acid concentration of the hot acid leach solution to about 15 g/L H_2SO_4 so as to minimize the neutralization requirements in the iron removal step;

to reduce the bulk of the ferric iron in solution to the ferrous state so as to minimize the sulphur dioxide consumption in the reducing leach step;

to increase the zinc concentration in solution by using zinc concentrate for neutralization of acid and reduction of iron;

to maintain sufficiently oxidizing conditions such that copper in solution is not precipitated as copper sulphide, which would then report to the zinc flotation concentrate; and

to produce a discharge slurry suitable for direct recovery of unreacted zinc sulphide as a flotation concentrate.

A two stage process was adopted with addition of zinc concentrate under oxidizing conditions to consume acid, followed by addition of concentrate under non-oxidizing conditions to reduce ferric iron. Six batch neutralization-reduction tests were performed to establish the feasibility of meeting all of the process objectives by this method, and to provide starting conditions for the continuous test. The continuous circuit was operated for 47.5 h, in concert with the continuous hot acid leach test. The continuous neutralization-reduction circuit was started 7.5 h after starting the hot acid leach circuit, when feed from the hot acid leach became available. 603 L of hot acid leach slurry and 77 kg of zinc concentrate were fed to the continuous neutralization-reduction circuit.

8.1 FEED MATERIALS

Hot acid leach slurry from the batch tests was treated in the neutralization-reduction batch tests. The concentrate used in the batch tests was surplus concentrate from another project, which was repulped to a 70% solids slurry for feeding to the reaction vessel. The repulp solution contained 50 g/L Zn so as not to dilute the process solution. HBM&S zinc concentrate was available for the continuous neutralization-reduction test. This concentrate was relatively coarse and was reground in a ball mill before repulping with demineralized water to a 70% solids slurry. In this case zinc was not added to the repulp solution because significant evaporation was anticipated in the continuous circuit. Analyses and particle size distributions for the concentrates are given in Section 5. The chemical analyses are summarized in the following table.

Zinc Concentrate	Analysis, %					
	Cd	Cu	Fe	Pb	S	Zn
Batch Tests	0.16	0.13	8.58	2.73	30.1	48.3
HBM&S (Continuous)	0.11	1.15	10.4	0.27	32.8	48.8

Discharge slurry from the hot acid leach circuit was used directly in the continuous neutralization-reduction circuit. The feed composition changed when the retention time in the hot acid leach circuit was doubled. The average feed compositions for the four periods of operation of the neutralization-reduction circuit are summarized below.

Component	Period	Analysis					
		Cu	Fe ²⁺	Fe	Pb	H ₂ SO ₄	Zn
Solution, g/L	1	3.11	2.3	13.5	-	51.8	60.0
	2	3.35	2.5	17.6	-	49.5	67.0
	3	3.04	2.6	19.6	-	48.0	69.2
	4	3.05	2.4	19.9	-	48.4	69.7
Solids, %	1	0.70	-	12.4	16.9	-	10.9
	2	0.65	-	10.8	17.8	-	9.9
	3	0.65	-	9.9	18.1	-	9.4
	4	0.65	-	9.7	21.6	-	8.1

8.2 BATCH TESTS

Six batch neutralization-reduction tests were carried out at 90°C in an autoclave under atmospheric conditions. The autoclave was used in preference to an open tank because of the superior oxygen dispersion achieved in this vessel. Oxygen was sparged into the slurry, with the sparge tube located below the lower, radial impeller. The upper impeller was an axial turbine to allow for good mixing of the slurry and to draw oxygen into the slurry from the vapour space.

The first three batch tests were conducted with the hot acid leach slurry produced with the first sample of oxide residue (Tables 8.1 to 8.3); the last three with the second sample (Tables 8.4 to 8.6). The results are summarized in the table overleaf.

In the first test all of the zinc concentrate was added at the start and oxygen was sparged into the vessel for the first four hours. Acid and iron solution concentrations during the test are plotted in Figure 8.1. The bulk of the ferric iron was reduced to the ferrous state within 30 minutes, but about half of the iron gradually reoxidized with oxygen. The acid concentration decreased to less than 20 g/L H₂SO₄ in the first two hours, resulting in some precipitation of ferric iron between 2 and 6 h. Shutting off the oxygen at 4 h had little

Test	Concentrate, kg/t *		Feed Solution			Discharge Solution		
	1st Stage	2nd Stage	Cu g/L	Fe ³⁺ g/L	H ₂ SO ₄ g/L	Cu g/L	Fe ³⁺ g/L	H ₂ SO ₄ g/L
1	253	-	5.6	16.3	43.0	5.3	1.5	12.8
2	189	59	5.5	17.1	38.5	5.4	1.3	15.2
3	150	-	5.8	18.2	35.7	5.9	10.8	13.4
4	243	-	6.1	19.2	51.5	6.4	13.2	14.5
5	200	96	6.0	20.8	45.1	5.5	4.2	9.0
6	127	98	6.0	21.1	43.3	5.8	5.7	9.8

* kg concentrate per tonne oxide residue

impact since most of the iron remaining in solution at this stage was in the ferrous state. It was significant that there was little precipitation of copper during the test.

Addition of zinc concentrate was staged in the second test, with about 25% of the concentrate added after shutting off the oxygen. As was the case for the first test, a significant quantity of the iron was reduced to the ferrous state in the early stages of the test. The acid concentration decreased to 10 g/L H₂SO₄ in the first 2 h and precipitation of iron was again evident, although somewhat slower than in the first test.

The concentrate addition in Test 3 was about 60% of that used in Tests 1 and 2. Acid neutralization was slower, and there was very little precipitation of iron. The solution contained 13.4 g/L H₂SO₄ and 10.8 g/L Fe³⁺ after 6 h with oxygen sparging throughout.

Test 4 was similar to Test 3 except that the second sample of oxide residue had been used to generate the feed slurry and the concentrate addition rate was increased to that of the first two tests. A large quantity of iron precipitated once the acid concentration was decreased to about 15 g/L H₂SO₄.

The concentrate addition was staged in Test 5, with about one third of the concentrate added after shutting off the oxygen supply. The terminal acid concentration was too low, however, and a portion of the iron was again precipitated from solution.

The concentrate addition rate was decreased to about 225 kg concentrate per tonne of oxide residue in Test 6, with about 55% of the concentrate added during the first stage with oxygen sparging. The acid concentration decreased to 19 g/L H₂SO₄ with little or no precipitation of iron (see Figure 8.2). Addition of the remaining concentrate after shutting off the oxygen resulted in consumption of a further 10 g/L H₂SO₄ and reduction of much of the remaining ferric iron in solution. The final solution contained 5.7 g/L Fe³⁺ and 9.8 g/L H₂SO₄. As was the case in all of the previous tests, very little copper was

precipitated from solution. Test 6 met with all of the process objectives and was used as the basis for startup of the continuous test.

8.3 CONTINUOUS NEUTRALIZATION-REDUCTION

8.3.1 Circuit Operation

A detailed description of the equipment and procedures for the neutralization-reduction circuit is provided in Section 6. The equipment flowsheet is provided in Figure 8.3. Hot acid leach discharge slurry was collected in a surge tank and pumped from the surge tank to the first of a series of five agitated stainless steel tanks arranged in cascade (NR1 to NR5). The operating volume of each tank was 13.5 L. Zinc concentrate slurry was pumped to the first, third and/or fourth tanks. Oxygen was sparged into the first three or four tanks. Heat was applied by means of steam coils in NR1 and NR3, and with heating tapes for NR2, NR4 AND NR5. Overflow slurry from NR5 was collected in pails.

Hot acid leach slurry was fed to NR1 as it was produced, and tanks NR2 to NR5 were allowed to fill with overflow slurry from the previous tank as it became available. No synthetic fill solutions or solids were used for the neutralization-reduction circuit.

8.3.2 Sampling Procedures

For control purposes, samples were collected from the first and last reaction tanks hourly. The control sample filtrates were titrated for Fe^{2+} and H_2SO_4 . Copper and total iron analyses were later determined by ICP. Profile samples were taken every six hours from the five reaction tanks. The profile solutions were titrated for Fe^{2+} and H_2SO_4 and were subsequently analyzed for As, Cd, Ca, Cu, Fe and Zn. Profile solids samples were analyzed for As, Cd, Ca, Cu, Fe, Pb, SiO_2 and Zn.

8.3.3 History of Events

Run Time h	Events and Changes
0	Started hot acid leach circuit. All tanks in neutralization-reduction circuit are empty.
5.8	Started feeding NR1 with hot acid leach discharge slurry (300 mL/min) and zinc concentrate slurry (21 g/min).
6.0	Applied steam and oxygen to NR1.
6.25	Began water feed to NR1, 4 mL/min.

Run Time h	Events and Changes
6.4	NR1 full, diverted feed to neutralization-reduction feed tank. Zinc concentrate slurry shut off. Operating NR1 in batch mode.
7.5	Sufficient surge collected. Started feed to NR1 from feed tank. Zinc concentrate slurry feed to NR1 restarted (21 g/min). Slurry allowed to overflow from NR1 to NR2.
8.0	Started oxygen (3 L/min) and water (4 mL/min) to NR2.
9.2	Started oxygen (2 L/min) and water (4 mL/min) to NR3, and concentrate to NR4 (13 g/min).
10.0	Started water to NR4 (4 mL/min).
10.6	Started collecting discharge from NR5 in pails. Started water to NR5 (4 mL/min).
11.67	Increased oxygen flow in NR1, NR2 and NR3 to 10, 6 and 4 L/min respectively. Increased zinc concentrate slurry feed rate to NR1 to 42 g/min.
13.0	Plug in NR1 zinc concentrate slurry line cleared.
14.33	Increased oxygen flow in NR2 and NR3 to 10 L/min and 5 L/min, respectively. Started adding oxygen to NR4 at 5 L/min. Started zinc concentrate slurry feed to NR3 (12.6 g/min).
15.0	Increased agitation in NR1 and NR5 to 1050 rev/min. Decreased concentrate slurry feed rate to NR1 to 25 g/min and increased concentrate slurry feed rate to NR3 to 25 g/min.
25.0	Decreased all feed rates by 50%, except oxygen, which remained the same.
32.33	Shut off all feeds to NR1. 4.4 L zinc hydroxide sludge, P.D. 1190 g/L, 24% solids, added to NR1.
32.5	Acid concentration in NR1 is 11.9 g/L H ₂ SO ₄ . All feeds restored.
36.0	Plug in zinc concentrate line cleared.

Run Time h	Events and Changes																		
37.5	Water addition rate increased to 6 mL/min per tank.																		
43.0	Decreased all oxygen flow rates by 50%.																		
49.0	Decreased concentrate slurry flow rates in NR1 and NR3 to 6.5 g/min.																		
55.0	Shutdown. Shut off feeds and heat, collected contents of each tank separately.																		
	<table><tr><td>Tank</td><td>Slurry Weight, kg</td><td>P.D., g/L</td></tr><tr><td>NR 1</td><td>18.0</td><td>1375</td></tr><tr><td>NR 2</td><td>19.4</td><td>1387</td></tr><tr><td>NR 3</td><td>19.7</td><td>1431</td></tr><tr><td>NR 4</td><td>18.6</td><td>1426</td></tr><tr><td>NR 5</td><td>19.2</td><td>1396</td></tr></table>	Tank	Slurry Weight, kg	P.D., g/L	NR 1	18.0	1375	NR 2	19.4	1387	NR 3	19.7	1431	NR 4	18.6	1426	NR 5	19.2	1396
Tank	Slurry Weight, kg	P.D., g/L																	
NR 1	18.0	1375																	
NR 2	19.4	1387																	
NR 3	19.7	1431																	
NR 4	18.6	1426																	
NR 5	19.2	1396																	

8.3.4 Operating Parameters

The operating parameters for the neutralization-reduction circuit are given in Tables 8.7 and 8.8, and are summarized below.

Period	Run Time (h)	Temp °C	Retention Time (h)	Oxygen L/min	Concentrate Addition	
					g/L Solution	kg/t Oxide Residue
1	8 to 25	89	3.3	30	130	470
2	25 to 43	88	7.1	30	158	630
3	43 to 49	89	6.4	15	159	650
4	49 to 55	89	6.3	15	94	390

Initially, hot acid leach slurry was fed to the first tank of the neutralization-reduction circuit at 310 mL/min, to keep pace with the rate of production of this slurry. Concentrate was fed to the first and fourth tanks, and oxygen was sparged into the first three tanks. However, the concentration of acid decreased by only 10 g/L H_2SO_4 , and the concentrate and oxygen flow rates were increased at 11.7 h run time. After 14 h the agitator tip speed and oxygen feed rates were increased and oxygen was introduced into the fourth tank in an attempt to further increase oxygen dispersion. Concentrate addition was also split between NR1, NR3 and NR4. After 25 h (the end of Period 1), the feed rates were stabilized at about 280 mL/min of hot acid leach slurry, 65 g/min of concentrate slurry and 30 L/min of oxygen.

The slurry feed rates were reduced by 50% in Period 2, and the oxygen maintained at 30 L/min. The oxygen flow rate was decreased to 15 L/min in Period 3. The concentrate addition rate was decreased by about 40% in Period 4.

8.3.5 Neutralization

A major objective of the neutralization-reduction circuit is to neutralize acid so as to minimize the reagent consumption and waste solids production in the iron removal circuit. Acid concentration in the first and last tanks of the neutralization-reduction circuit was monitored hourly. Acid, ferrous iron, total iron and copper analyses for these control samples are given in Tables 8.9 and 8.10, and the NR5 analyses are plotted against run time in Figures 8.4 and 8.5. Iron and acid concentrations are summarized below.

Period	Run Time h	Feed			NR5			H ₂ SO ₄ Consump g/L	Equiv. Acid Consump g/L
		Fe ²⁺ g/L	Fe g/L	H ₂ SO ₄ g/L	Fe ²⁺ g/L	Fe g/L	H ₂ SO ₄ g/L		
1	8 to 25	2.3	13.5	51.8	13.2	15.8	32.6	19.2	26.7
2	25 to 43	2.5	17.6	49.5	14.3	19.7	21.0	28.5	37.0
3	43 to 49	2.6	19.6	48.0	15.4	21.1	18.1	29.9	39.8
4	49 to 55	2.4	19.9	48.4	14.4	21.7	18.8	29.6	38.5

The concentration of acid in the last tank of the neutralization-reduction circuit was 40 g/L H₂SO₄ at the start of the run, compared with 52 g/L H₂SO₄ in the feed (Figure 8.5). Several changes to the operation of the circuit were made during Period 1 in an attempt to increase the amount of acid neutralized (see Section 8.3.4). The acid concentration in NR5 decreased to 30 g/L H₂SO₄ as a result of these changes, but this concentration was still significantly higher than the target of 15 g/L H₂SO₄.

Examination of the solution profiles for the circuit (Tables 8.11 and 8.12) revealed that some benefit could be obtained with a longer retention time, and thus the retention time was doubled after 25 hours of operation. This change resulted in a further increase in the acid consumption, with only 18 g/L H₂SO₄ remaining in solution after 37 hours operation. This acid concentration was considered adequate since some consumption of acid was anticipated in the reducing leach step, and a further increase in retention time to neutralize additional acid in the neutralization-reduction step was therefore considered to be unnecessary. An acid concentration of 17 to 18 g/L H₂SO₄ was maintained when the oxygen feed rate was decreased by 50% after 43 h, but the acid concentration began to increase again when the concentrate feed rate was decreased by 40% after 49 h operation (Figure 8.5).

A concentrate addition rate of 225 kg/t oxide residue was required in the batch tests to produce a discharge solution with sufficiently low ferric iron and acid concentrations.

The optimum concentrate addition rate in the continuous test was between 390 and 650 kg/t oxide residue. The lower rate was sufficient in the batch tests since fortified spent electrolyte was used in the hot acid leach batch tests and a relatively small volume of solution was therefore treated in neutralization-reduction.

An attempt was made to decrease the concentrate requirement in the continuous test after 32 h. The acid concentration in the first tank was reduced by rapid addition of zinc hydroxide sludge after the feeds to the circuit were shut off (see Section 8.3.3). It was hoped that the lower acid concentration might result in a more rapid ferrous iron oxidation and therefore a greater acid neutralization in this stage. The lower acid concentration was not maintained once the feeds were restored, however, and the circuit returned to the previous conditions.

8.3.6 Reduction

The reduction of ferric to ferrous iron limits the precipitation of iron when the acid concentration is decreased, and also reduces reagent consumption in the reducing leach step. Some ferric iron is required, however, to prevent removal of copper from the solution by a metathesis reaction with the zinc sulphide concentrate (see Section 4). The target ferric iron concentration was 5 g/L in the NR5 solution.

Iron concentration profiles for the continuous test are given in Tables 8.11 and 8.12 and are summarized below.

Period	Run Time, h	Fe ³⁺ Analysis, g/L					
		Feed	NR1	NR2	NR3	NR4	NR5
1	8 to 25	11.2	3.5	5.0	3.5	3.5	2.6
2	25 to 43	15.1	10.3	10.8	8.3	9.6	5.4
3	43 to 49	17.0	8.4	9.5	6.7	7.7	5.7
4	49 to 55	17.5	12.2	13.2	10.9	9.9	7.3

The concentration of ferric iron in the feed solution increased significantly after the first period of operation, when the retention time and the acid addition rate were increased in the hot acid leach circuit (see Section 7.3 and Figure 8.6). Since the target concentration was 5 g/L Fe³⁺ in NR5, there was twice as much ferric iron to reduce in the latter periods compared with the first period.

The bulk of the ferric iron was reduced in NR1 during the first period of operation, and there was sufficient excess zinc concentrate present to prevent a net reoxidation of iron in the remainder of the circuit. The concentration of ferric iron ranged from 2.6 to 5.0 g/L in all of the tanks.

Increasing the retention time in the neutralization-reduction circuit in Period 2 resulted in greater reoxidation of ferrous iron by oxygen and the concentration of ferric iron was about 10 g/L in NR1 to NR4 (with oxygen sparging). Oxygen was not added to NR5, and the iron concentration decreased to the target level of 5 g/L Fe^{3+} . The ferric iron concentration was abnormally high between 37 and 40 h, likely as a result of feed interruptions at 32 h and 36 h (see Section 8.3.3).

A lower oxygen flow in Period 3 resulted in lower ferric iron concentrations in NR1 to NR4. The NR5 concentration remained at about 5 g/L Fe^{3+} .

A lower concentrate feed rate in Period 4 resulted in significantly higher ferric iron concentrations in all of the tanks. The NR5 concentration was 7.3 g/L Fe^{3+} on average, increasing throughout the period to 9.3 g/L at the end (see Figure 8.6). Insufficient concentrate was added during this period to meet either the iron reduction or the acid consumption objectives.

Although it was originally intended that a portion of the concentrate would be added under non-oxidizing conditions, as was the case in the batch tests, the slow neutralization of acid in the early stages of the continuous test dictated that oxygen be fed to NR4, where the last concentrate addition was made. Reduction of ferric iron proved to be rapid with or without oxygen sparging, however, and a separate addition of concentrate to NR5 did not prove necessary.

8.3.7 Zinc Extraction

Extraction of zinc from the zinc concentrate added in the neutralization-reduction circuit has been estimated from the solids analyses and rates, in the table following.

Period	Run Time h	Ret. Time h	HAL Solids		Concentrate		NR5 Solids		Zinc Ext'n* %
			Rate g/h	Zn %	Rate g/h	Zn %	Rate g/h	Zn %	
1	8 to 25	3.3	3962	10.9	2269	48.8	5666	24.1	15.7
2	25 to 43	7.1	1652	9.9	1329	48.8	2666	19.1	46.7
3	43 to 49	6.4	1684	9.4	1371	48.8	2636	16.5	58.6
4	49 to 55	6.3	1451	8.1	819	48.8	2019	11.9	69.3

* Assuming extraction from concentrate alone.

Little emphasis should be placed on the absolute values of these extractions since the operating periods were short and the retention times in the circuit were relatively long. Zinc extraction on average was between 40 and 50%. Increasing the retention time in Period 2 had a significant influence, and a retention time of about 6 h appears to be

required to achieve the process objectives as well as a zinc extraction of 50% or greater. A large excess of concentrate was used during most of the run, and the zinc extraction only approached 70% in the last period of operation, when the concentrate feed was reduced to about 390 kg/t oxide residue. Optimization of concentrate addition would likely result in a zinc extraction between 60 and 70%.

8.3.8 Copper Deportment

Efficient recovery of copper in the process requires that copper extracted in the hot acid leach step remains in solution during the neutralization-reduction step. Any copper that is precipitated via metathesis with sphalerite during this step would subsequently report to the zinc flotation concentrate, and decrease the recovery of copper to the copper flotation concentrate. Solution and solids analyses for copper in neutralization-reduction are given in Tables 8.9 to 8.14, and control solution analyses are plotted in Figure 8.4. Average analyses for the four operating periods are summarized below.

Period	Run Time h	Copper Analysis				
		Solution, g/L		Solids, %		
		Feed	NR5	Feed (HAL)	Feed (Con)	NR5
1	8 to 25	3.11	3.24	0.70	1.15	0.84
2	25 to 43	3.35	3.79	0.65	1.15	0.77
3	43 to 49	3.04	3.58	0.65	1.15	0.72
4	49 to 55	3.05	3.29	0.65	1.15	0.68

During the continuous test there was a small net extraction of copper owing to reaction of copper contained in the zinc concentrate added to the neutralization-reduction circuit; there was no evidence of precipitation of copper at any time. Favourable copper deportment in the circuit can be accounted for by the oxidizing conditions that were maintained in all five tanks. Although oxygen was not sparged into the last tank of the train to allow for reduction of ferric iron, the concentration of ferric iron in solution was not allowed to fall below about 2 g/L, and averaged about 5 g/L (see Figure 8.6). It may be possible to operate with a lower ferric iron concentration and still keep copper in solution. This would result in a lower reagent consumption in the reducing leach step.

8.4 CONCLUSIONS AND RECOMMENDATIONS

Addition of zinc concentrate to the hot acid leach slurry while sparging oxygen is effective in neutralizing excess acid in the hot acid leach solution. A retention time of 6 h at 90°C is sufficient to reduce the concentration of acid in the solution from about 50 g/L to between 15 and 20 g/L H₂SO₄.

By limiting the quantity of oxygen that is fed to the system, ferric iron in the solution may be reduced to the ferrous state, which is desirable to keep iron in solution and to reduce reagent requirements in the reducing leach step.

The zinc concentrate addition rate was not optimized during the continuous test, but it is estimated that about 400 kg of concentrate per tonne of stockpiled oxide residue is required to produce a solution containing 15 g/L H_2SO_4 and 5 g/L ferric iron.

A further objective of the neutralization-reduction step is to maintain the conditions sufficiently oxidizing that copper extracted into the solution during the hot acid acid leach step is not precipitated during neutralization-reduction. Control of the operating conditions did not prove difficult during the continuous test, and a small net extraction of copper was observed.

Table 8.1 Neutralization-Reduction: Batch Test 1

Charge	:	2.0 L hot acid leach slurry (blend of Batch Tests 1 to 3); 196 g zinc concentrate as 70% solids slurry, injected at temperature
Temperature	:	90°C
Pressure	:	Atmospheric
Oxidant	:	Oxygen, 0.5 L/min sparge, between 0 and 4 h
Equipment	:	3.8 L titanium-lined autoclave, equipped with baffles
Agitation	:	Axial over radial impellers, tip speed 4.7 m/s

	Head		Retention Time, h				
	Slurry	Con	0.5	1.0	2.0	4.0	6.0
Solutions, g/L							
As	5.50	n.a.	4.14	3.94	3.30	1.21	0.92
Cd	0.58	n.a.	0.55	0.56	0.58	0.61	0.58
Ca	0.51	n.a.	0.53	0.55	0.60	0.60	0.60
Cu	5.58	n.a.	5.10	5.18	5.56	5.50	5.28
Fe ²⁺	3.3	n.a.	14.8	14.8	9.2	7.6	7.0
Fe	19.6	n.a.	19.0	19.4	20.4	11.1	8.48
H ₂ SO ₄	43.0	n.a.	29.8	24.8	17.6	11.6	12.8
Zn	52.6	50	61.4	64.8	74.4	86.4	85.6
Solids, %							
As	2.03	0.11	2.33	2.43	2.36	3.15	3.17
Cd	0.06	0.16	0.09	0.09	0.09	0.10	0.11
Ca	5.68	0.47	6.17	6.24	5.77	3.46	2.06
Cu	0.62	0.13	0.67	0.70	0.63	0.78	0.87
Fe	7.84	8.58	9.44	9.45	8.89	12.4	13.9
Pb	15.4	2.73	16.6	16.6	15.6	18.7	20.4
SiO ₂	3.85	3.76	4.58	4.55	4.06	3.73	4.44
S(SO ₄)	8.12	0.26	n.a.	n.a.	n.a.	n.a.	5.01
S	10.1	30.1	14.2	13.4	13.3	12.9	12.1
Zn	4.85	48.3	12.0	11.6	10.3	10.6	10.8

n.a. = not analyzed

Table 8.2 Neutralization-Reduction: Batch Test 2

Charge	:	2.0 L hot acid leach slurry (blend of Batch Tests 1 to 3); 192 g zinc concentrate as 70% solids slurry, 146 g injected at temperature, 46 g at 4 h
Temperature	:	90°C
Pressure	:	Atmospheric
Oxidant	:	Oxygen, 0.5 L/min sparge, between 0 and 4 h
Equipment	:	3.8 L titanium-lined autoclave, equipped with baffles
Agitation	:	Axial over radial impellers, tip speed 4.7 m/s

	Head		Retention Time, h					
	Slurry	Con	0.5	1.0	2.0	4.0	5.0	6.0
Solutions, g/L								
As	5.38	n.a.	4.68	4.64	4.06	1.84	0.78	0.44
Cd	0.58	n.a.	0.58	0.61	0.62	0.61	0.59	0.64
Ca	0.48	n.a.	0.58	0.56	0.54	0.53	0.56	0.60
Cu	5.50	n.a.	5.38	5.50	5.48	5.46	5.22	5.38
Fe ²⁺	3.5	n.a.	12.2	14.2	6.0	11.0	11.4	5.4
Fe	20.6	n.a.	21.0	21.6	21.0	18.1	13.9	6.66
H ₂ SO ₄	38.5	n.a.	27.8	24.6	10.2	8.6	8.6	15.2
Zn	53.3	50	62.8	66.0	74.0	79.6	81.0	89.4
Solids, %								
As	2.03	0.11	2.42	2.48	2.67	2.92	2.83	2.87
Cd	0.06	0.16	0.09	0.09	0.09	0.09	0.09	0.08
Ca	5.68	0.47	6.45	6.50	6.13	6.35	5.83	5.78
Cu	0.62	0.13	0.70	0.70	0.72	0.70	0.67	0.68
Fe	7.84	8.58	9.44	9.32	9.88	10.0	10.3	11.2
Pb	15.4	2.73	17.2	17.2	18.0	17.5	16.4	16.0
SiO ₂	3.85	3.76	4.61	4.49	4.72	4.67	4.35	4.22
S(SO ₄)	8.12	0.26	n.a.	n.a.	n.a.	n.a.	n.a.	6.88
S	10.1	30.1	n.a.	n.a.	n.a.	n.a.	n.a.	14.1
Zn	4.85	48.3	10.6	10.3	9.14	8.00	9.66	9.04

n.a. = not analyzed

Table 8.3 Neutralization-Reduction: Batch Test 3

Charge	:	2.0 L hot acid leach slurry (blend of Batch Tests 1 to 3); 116 g zinc concentrate as 70% solids slurry, injected at temperature
Temperature	:	90°C
Pressure	:	Atmospheric
Oxidant	:	Oxygen, 0.5 L/min sparge
Equipment	:	3.8 L titanium-lined autoclave, equipped with baffles
Agitation	:	Axial over radial impellers, tip speed 4.7 m/s

	Head		Retention Time, h				
	Slurry	Con	0.5	1.0	2.0	4.0	6.0
Solutions, g/L							
As	5.72	n.a.	5.14	4.52	4.26	4.22	3.88
Cd	0.51	n.a.	0.75	0.54	0.52	0.57	0.60
Ca	0.60	n.a.	0.78	0.57	0.54	0.60	0.64
Cu	5.80	n.a.	5.82	5.28	5.10	5.52	5.86
Fe ²⁺	3.7	n.a.	13.0	12.6	11.8	11.8	11.6
Fe	21.9	n.a.	22.6	20.6	19.8	21.4	22.4
H ₂ SO ₄	35.7	n.a.	28.6	25.6	21.6	19.4	13.4
Zn	54.9	50	64.2	59.8	59.4	67.6	75.4
Solids, %							
As	2.03	0.11	2.59	2.85	2.59	2.80	3.12
Cd	0.06	0.16	0.09	0.10	0.09	0.09	0.10
Ca	5.68	0.47	6.74	6.90	6.78	6.92	7.06
Cu	0.62	0.13	0.75	0.78	0.76	0.76	0.79
Fe	7.84	8.58	9.77	9.92	9.73	9.88	10.1
Pb	15.4	2.73	18.1	18.5	18.1	18.4	18.9
SiO ₂	3.85	3.76	5.19	5.73	5.03	5.06	5.98
S(SO ₄)	8.12	0.26	n.a.	n.a.	n.a.	n.a.	8.51
S	10.1	30.1	n.a.	n.a.	n.a.	n.a.	12.5
Zn	4.85	48.3	10.0	10.1	9.54	9.04	8.56

n.a. = not analyzed

Table 8.4 Neutralization-Reduction: Batch Test 4

Charge	:	2.0 L hot acid leach slurry (Batch Test 4); 187.5 g zinc concentrate as 70% solids slurry, injected at temperature; 0.1 L wash water
Temperature	:	90°C
Pressure	:	Atmospheric
Oxidant	:	Oxygen, 0.5 L/min sparge
Equipment	:	3.8 L titanium-lined autoclave, equipped with baffles
Agitation	:	Axial over radial impellers, tip speed 4.7 m/s

	Head		Retention Time, h				
	Slurry	Con	0.5	1.0	2.0	4.0	6.0
Solutions, g/L							
As	5.41	n.a.	5.22	5.29	5.06	4.90	3.35
Cd	0.24	n.a.	0.28	0.29	0.30	0.32	0.32
Ca	0.65	n.a.	0.47	0.56	0.54	0.52	0.54
Cu	6.10	n.a.	6.44	6.55	6.39	6.46	6.39
Fe ²⁺	5.8	n.a.	20.0	18.3	16.8	15.5	7.7
Fe	25.0	n.a.	30.7	31.5	31.0	31.6	20.9
H ₂ SO ₄	51.5	n.a.	36.0	31.8	24.5	15.6	14.5
Zn	61.6	50	79.6	83.2	84.4	90.9	98.1
Solids, %							
As	0.45	0.11	0.49	0.48	0.52	0.58	0.90
Cd	0.10	0.16	0.10	0.10	0.10	0.10	0.10
Ca	7.33	0.47	6.77	7.01	7.01	7.23	6.68
Cu	0.52	0.13	0.41	0.42	0.42	0.42	0.43
Fe	11.8	8.58	10.2	10.4	10.5	10.5	12.5
Pb	19.6	2.73	16.7	17.0	17.5	17.6	16.7
SiO ₂	6.34	3.76	5.95	6.10	6.22	6.17	5.93
Zn	10.8	48.3	14.5	14.3	13.0	12.0	9.83

n.a. = not analyzed

Table 8.5 Neutralization-Reduction: Batch Test 5

Charge	:	2.0 L hot acid leach slurry (Batch Test 5); 284.2 g zinc concentrate as 70% solids slurry, 192 g injected at temperature, 92.2 g at 3 h; 0.1 L wash water
Temperature	:	90°C
Pressure	:	Atmospheric
Oxidant	:	Oxygen, 0.5 L/min sparge, between 0 and 3 h
Equipment	:	3.8 L titanium-lined autoclave, equipped with baffles
Agitation	:	Axial over radial impellers, tip speed 4.7 m/s

	Head		Retention Time, h						
	Slurry	Con	0.5	1.0	2.0	3.0	4.0	5.0	6.0
Solutions, g/L									
As	5.29	n.a.	4.35	4.45	4.48	4.75	1.59	1.37	1.12
Cd	0.24	n.a.	0.25	0.27	0.28	0.31	0.24	0.26	0.25
Ca	0.42	n.a.	0.50	0.52	0.52	0.50	0.54	0.55	0.54
Cu	6.05	n.a.	5.68	5.94	6.06	6.54	5.31	5.59	5.47
Fe ²⁺	5.8	n.a.	18.9	17.3	16.6	15.8	20.2	20.0	19.0
Fe	26.6	n.a.	26.4	27.6	28.5	31.0	23.8	24.8	23.2
H ₂ SO ₄	45.1	n.a.	34.1	28.6	22.0	15.5	11.0	9.6	9.0
Zn	61.7	50	69.0	73.5	78.6	87.8	76.8	81.6	81.0
Solids, %									
As	0.32	0.11	0.47	0.49	0.59	0.65	1.20	1.28	1.27
Cd	0.09	0.16	0.10	0.10	0.10	0.10	0.11	0.11	0.11
Ca	7.44	0.47	6.87	6.66	6.87	6.72	5.88	6.06	5.89
Cu	0.48	0.13	0.45	0.44	0.44	0.44	0.42	0.42	0.41
Fe	10.7	8.58	10.8	10.9	10.7	10.8	11.1	11.0	11.1
Pb	18.4	2.73	17.3	17.0	16.7	17.1	15.8	15.6	15.3
SiO ₂	5.95	3.76	6.57	6.47	6.35	6.49	6.32	6.30	6.08
Zn	9.82	48.3	12.9	13.8	13.2	12.1	15.1	14.7	14.4

n.a. = not analyzed

Table 8.6 Neutralization-Reduction: Batch Test 6

Charge	:	2.0 L hot acid leach slurry (Batch Test 5); 174 g zinc concentrate as 70% solids slurry, 98 g injected at temperature, 76 g at 3 h; 0.1 L wash water
Temperature	:	90°C
Pressure	:	Atmospheric
Oxidant	:	Oxygen, 0.5 L/min sparge, between 0 and 3 h
Equipment	:	3.8 L titanium-lined autoclave, equipped with baffles
Agitation	:	Axial over radial impellers, tip speed 4.7 m/s

	Head		Retention Time, h					
	Slurry	Con	0.5	1.0	2.0	3.0	4.0	5.0
Solutions, g/L								
As	5.42	n.a.	4.74	4.84	5.00	4.61	2.11	1.68
Cd	0.26	n.a.	0.28	0.29	0.31	0.30	0.26	0.27
Ca	0.42	n.a.	0.55	0.52	0.52	0.49	0.56	0.49
Cu	6.04	n.a.	5.93	6.08	6.38	5.95	5.46	5.76
Fe ²⁺	5.9	n.a.	17.5	16.7	15.4	14.0	19.4	19.5
Fe	27.0	n.a.	27.6	28.4	29.8	28.1	24.7	25.2
H ₂ SO ₄	43.3	n.a.	31.8	28.4	23.6	19.0	12.2	9.8
Zn	61.5	50	72.2	75.5	81.0	78.0	78.1	83.2
Solids, %								
As	0.42	0.11	0.48	0.54	0.59	0.55	1.02	1.11
Cd	0.10	0.16	0.10	0.10	0.10	0.10	0.11	0.11
Ca	7.93	0.47	6.65	6.74	6.97	7.11	6.03	5.71
Cu	0.50	0.13	0.43	0.44	0.44	0.44	0.40	0.39
Fe	11.0	8.58	10.3	10.5	10.5	10.7	10.7	10.4
Pb	19.8	2.73	17.3	17.5	17.8	18.2	16.0	15.2
SiO ₂	6.50	3.76	6.08	6.20	6.25	6.45	6.12	6.00
Zn	10.3	48.3	13.1	12.8	11.9	11.6	15.2	14.8

n.a. = not analyzed

Table 8.7 Neutralization-Reduction Operating Parameters (0 to 36 h)

Run Time h	Slurry Feed mL/min	Water mL/min	Concentrate, g/min*			Oxygen, L/min				Temp °C ⁺	Discharge	
			NR1	NR3	NR4	NR1	NR2	NR3	NR4		Rate mL/min	P.D. g/L
8	308	4	20.4	—	—	5	—	—	—	88	—	—
9	313	8	20.1	—	—	5	3	—	—	92	—	—
10	311	12	19.9	—	13.4	5	3	2	—	92	—	—
11	312	25	20.1	—	13.4	5	3	2	—	90	344	1395
12	315	25	42.3	—	13.5	10	6	4	—	86	375	1375
13	320	25	42.3	—	13.6	10	6	4	—	90	286	1361
14	315	25	42.3	—	13.6	10	6	4	—	89	233	1380
15	310	25	26.0	25.3	13.4	10	10	5	5	89	339	1362
16	315	25	26.0	25.3	13.3	10	10	5	5	88	256	1385
17	310	25	25.8	24.9	13.2	10	10	5	5	89	331	1407
18	310	25	25.6	24.5	13.3	10	10	5	5	88	312	1415
19	310	25	23.7	24.4	13.0	10	10	5	5	89	328	1412
20	310	25	25.0	24.5	13.0	10	10	5	5	88	319	1385
21	310	25	25.7	24.8	13.1	10	10	5	5	89	309	1432
22	310	25	25.0	24.2	13.0	10	10	5	5	89	315	1429
23	285	24	25.8	25.0	13.3	10	10	5	5	89	297	1422
24	285	24	25.8	25.0	13.4	10	10	5	5	89	293	1439
25	282	26	25.6	25.2	13.8	10	10	5	5	88	288	1440
26	133	13	12.3	12.6	6.6	10	10	5	5	89	155	1454
27	142	13	12.5	13.0	6.4	10	10	5	5	90	155	1454
28	147	13	12.7	13.1	5.8	10	10	5	5	88	131	1458
29	148	14	12.5	12.4	5.7	10	10	5	5	87	131	1458
30	150	14	12.5	12.7	6.4	10	10	5	5	87	119	1446
31	146	14	12.4	12.8	6.2	10	10	5	5	88	119	1446
32	146	14	13.1	13.2	6.5	10	10	5	5	88	130	1485
33	145	14	12.8	13.5	6.3	10	10	5	5	87	130	1485
34	150	14	12.4	13.0	6.5	10	10	5	5	88	130	1526
35	147	14	12.8	13.4	6.1	10	10	5	5	87	130	1526
36	149	15	12.0	13.7	6.5	10	10	5	5	88	130	1524

* 70% solids slurry

+ Mean value for five tanks NR1 to NR5

Table 8.8 Neutralization-Reduction Operating Parameters (37 to 55 h)

Run Time h	Slurry Feed mL/min	Water mL/min	Concentrate, g/min*			Oxygen, L/min				Temp °C+	Discharge	
			NR1	NR3	NR4	NR1	NR2	NR3	NR4		Rate mL/min	P.D. g/L
37	152	15	12.7	12.0	6.6	10	10	5	5	88	130	1524
38	150	30	12.8	12.1	6.6	10	10	5	5	88	166	1514
39	150	30	12.8	11.9	6.8	10	10	5	5	88	166	1514
40	150	30	12.3	11.8	6.5	10	10	5	5	88	145	1513
41	148	30	12.3	13.1	6.5	10	10	5	5	87	145	1513
42	150	30	12.0	12.7	6.3	10	10	5	5	87	142	1479
43	150	30	13.0	12.8	6.3	10	10	5	5	88	142	1479
44	150	30	13.0	12.8	6.4	5	5	2.5	2.5	88	139	1479
45	150	31	13.3	12.7	6.3	5	5	2.5	2.5	89	139	1479
46	150	31	12.9	12.8	6.4	5	5	2.5	2.5	88	156	1481
47	146	30	12.7	12.5	6.2	5	5	2.5	2.5	89	156	1481
48	150	30	13.2	13.1	6.5	5	5	2.5	2.5	89	151	1400
49	155	30	13.8	14.2	7.0	5	5	2.5	2.5	89	151	1400
50	148	30	6.7	6.7	6.8	5	5	2.5	2.5	89	160	1469
51	148	30	6.4	6.6	6.7	5	5	2.5	2.5	89	160	1469
52	150	30	6.2	6.4	6.5	5	5	2.5	2.5	89	156	1439
53	152	30	6.4	7.2	6.8	5	5	2.5	2.5	89	156	1439
54	152	30	6.1	6.8	6.7	5	5	2.5	2.5	89	165	1461
55	155	30	5.5	6.2	6.3	5	5	2.5	2.5	89	165	1461

* 70% solids slurry

+ Mean value for five tanks NR1 to NR5

Table 8.9 Neutralization-Reduction Control Solution Analyses (0 to 37 h)

Run Time h	Analysis, g/L							
	NR1				NR5			
	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Cu	Fe ²⁺	Fe	H ₂ SO ₄
9*	3.60	10.0	15.5	45.4	n.s.	n.s.	n.s.	n.s.
10**	3.41	9.5	14.8	44.8	n.s.	n.s.	n.s.	n.s.
11**	3.27	7.5	14.0	46.3	3.48	13.3	16.2	40.5
12**	3.13	7.4	13.6	44.9	3.09	11.7	14.2	38.5
13**	3.25	11.3	14.8	45.7	3.28	13.3	15.5	42.6
14**	2.66	8.1	12.1	33.5	2.71	11.8	13.0	33.2
16**	3.23	13.7	15.4	36.6	3.17	13.5	15.3	33.4
17	3.11	10.8	14.2	46.5	3.22	13.2	15.7	30.7
18	3.05	12.1	14.1	44.5	2.99	12.6	14.7	28.5
19	3.35	11.2	15.7	48.0	3.30	13.3	16.5	30.2
20	3.33	11.2	15.3	49.2	3.18	13.1	15.7	30.0
21	3.44	11.6	15.8	48.2	3.23	12.9	16.0	29.5
22	3.17	10.2	14.5	44.0	3.36	13.9	16.7	30.0
23	3.39	11.9	15.6	47.0	3.38	13.5	16.7	30.5
24	3.29	10.5	15.5	39.8	3.43	13.9	17.4	30.0
25	3.51	11.7	16.6	46.0	3.57	14.1	18.1	29.1
26	3.47	11.3	16.6	45.9	3.47	14.5	17.8	28.0
27	3.39	11.7	16.4	42.8	3.37	12.6	17.3	23.8
29	3.61	7.1	16.9	42.3	3.47	14.2	17.6	24.0
30	3.56	11.2	17.0	46.5	3.44	13.0	17.4	24.5
31	3.88	12.3	19.0	46.8	3.87	14.8	20.0	23.8
32	3.46	11.1	16.8	42.8	3.89	15.5	19.8	25.0
33	3.37	9.5	15.8	23.8	3.73	14.8	19.1	22.6
34	3.43	9.5	16.5	35.3	3.69	14.1	18.9	21.5
35	3.33	10.7	16.2	38.5	3.94	15.5	20.1	22.5
36	3.67	8.6	18.2	44.5	3.32	13.0	17.0	16.3
37	3.73	9.5	19.6	44.7	3.81	14.0	19.9	17.9

* NR2 rather than NR1

** NR3 rather than NR1

n.s. = no sample

Table 8.10 Neutralization-Reduction Control Solution Analyses (38 to 55)

Run Time h	Analysis, g/L							
	NR1				NR5			
	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Cu	Fe ²⁺	Fe	H ₂ SO ₄
38	4.24	10.7	22.4	41.0	4.56	15.2	23.2	18.2
39	3.66	10.5	19.9	41.7	4.94	14.2	25.4	17.5
40	4.68	11.8	26.3	42.2	3.93	14.2	20.5	17.7
41	3.69	11.6	21.2	42.0	3.74	14.6	19.8	18.5
42	3.73	11.9	21.8	43.7	3.87	14.9	20.8	18.2
43	4.21	11.8	25.8	41.0	3.46	14.1	19.7	16.5
44	3.34	9.4	20.0	41.5	3.77	15.5	21.0	18.6
45	3.30	11.5	20.2	41.5	3.59	14.6	20.4	18.1
46	3.31	11.3	19.9	40.0	3.52	15.0	20.4	18.2
47	3.16	12.1	19.8	39.8	3.49	15.3	20.7	18.3
48	3.16	12.5	20.0	39.3	3.58	15.8	21.7	18.0
49	3.15	12.1	20.5	36.5	3.52	16.2	22.3	17.5
50	3.00	10.9	19.6	37.1	3.39	16.6	21.9	17.5
51	3.07	8.0	20.0	39.8	3.36	16.0	22.1	16.8
52	3.02	7.3	19.8	41.6	3.25	13.9	21.5	18.0
53	2.92	7.5	19.0	37.8	3.16	13.5	20.9	19.3
54	2.99	6.2	19.5	38.0	3.21	12.9	21.4	19.5
55	2.95	7.2	19.4	42.3	3.37	13.3	22.6	22.0

Table 8.11 Neutralization-Reduction Profile Solution Analyses (0 to 37 h)

Run Time h	Sample	Analysis, g/L							
		As	Cd	Ca	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Zn
13	Feed	2.56	0.12	0.56	2.90	2.3	12.1	51.8	56.3
	NR1	2.53	0.15	0.60	3.16	14.6	15.7	40.8	68.4
	NR2	2.56	0.14	0.62	3.18	12.0	14.8	43.7	66.5
	NR3	2.60	0.14	0.58	3.25	11.3	14.8	45.7	66.4
	NR4	2.58	0.14	0.65	3.24	14.1	15.6	42.2	67.9
	NR5	2.58	0.14	0.62	3.28	13.3	15.5	42.6	67.5
19	Feed	2.74	0.13	0.66	3.07	2.3	13.5	51.2	59.4
	NR1	2.76	0.15	0.72	3.35	11.2	15.7	48.0	68.2
	NR2	2.76	0.15	0.71	3.40	10.5	16.1	45.2	70.1
	NR3	2.55	0.15	0.64	3.25	13.2	16.0	38.0	70.3
	NR4	2.52	0.16	0.72	3.28	12.0	16.2	33.5	71.9
	NR5	2.48	0.16	0.68	3.30	13.3	16.5	30.2	73.9
25	Feed	3.01	0.14	0.66	3.36	2.4	14.8	52.5	64.4
	NR1	2.87	0.16	0.57	3.51	11.7	16.6	46.0	71.7
	NR2	2.88	0.16	0.56	3.54	10.3	17.0	41.8	72.6
	NR3	2.76	0.17	0.53	3.56	13.4	17.6	36.5	76.0
	NR4	2.67	0.17	0.61	3.48	12.7	17.4	33.3	75.8
	NR5	2.68	0.17	0.53	3.57	14.1	18.1	29.1	79.2
31	Feed	2.86	0.13	0.51	3.19	2.4	14.9	50.0	62.4
	NR1	3.15	0.17	0.60	3.88	12.3	19.0	46.8	79.3
	NR2	3.08	0.17	0.53	3.83	9.6	18.9	41.8	78.1
	NR3	2.96	0.18	0.50	3.86	13.9	19.5	33.8	83.1
	NR4	2.91	0.19	0.51	3.92	13.2	20.1	25.8	87.4
	NR5	2.83	0.19	0.48	3.87	14.8	20.0	23.8	87.3
37	Feed	3.16	0.15	0.61	3.51	2.5	18.8	50.2	71.6
	NR1	3.11	0.17	0.64	3.73	9.5	19.6	44.7	78.0
	NR2	3.14	0.18	0.54	3.87	8.7	19.7	38.8	83.0
	NR3	3.03	0.20	0.53	3.97	13.2	20.4	28.2	90.9
	NR4	3.06	0.22	0.55	4.20	12.5	21.8	20.0	97.8
	NR5	2.70	0.20	0.47	3.81	14.0	19.9	17.9	89.1

Table 8.12 Neutralization-Reduction Profile Solution Analyses (38 to 55 h)

Run Time h	Sample	Analysis, g/L							
		As	Cd	Ca	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Zn
43	Feed	3.71	0.18	0.74	4.27	2.6	26.3	48.2	92.0
	NR1	3.35	0.19	0.58	4.21	11.8	25.8	41.0	93.3
	NR2	3.01	0.18	0.50	3.90	11.1	23.2	36.7	85.5
	NR3	3.31	0.22	0.61	4.49	14.3	26.5	29.2	102
	NR4	3.23	0.23	0.62	4.59	13.9	26.6	21.0	106
	NR5	2.37	0.18	0.46	3.46	14.1	19.7	16.5	81.9
49	Feed	2.70	0.13	0.55	3.04	2.6	19.6	48.0	69.2
	NR1	2.51	0.14	0.54	3.15	12.1	20.5	36.5	74.6
	NR2	2.50	0.15	0.53	3.27	11.5	21.0	33.0	76.5
	NR3	2.34	0.16	0.56	3.20	14.0	20.7	25.0	77.4
	NR4	2.38	0.17	0.53	3.38	14.0	21.7	19.3	82.7
	NR5	2.43	0.18	0.50	3.52	16.2	22.3	17.5	86.6
55	Feed	2.72	0.13	0.57	3.05	2.4	19.9	48.4	69.7
	NR1	2.46	0.12	0.56	2.95	7.2	19.4	42.3	67.7
	NR2	2.56	0.13	0.56	3.13	7.5	20.7	39.0	71.9
	NR3	2.48	0.14	0.55	3.18	10.3	21.2	32.0	75.0
	NR4	2.36	0.15	0.55	3.20	11.6	21.5	25.1	77.5
	NR5	2.43	0.16	0.59	3.37	13.3	22.6	22.0	82.6

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Table 8.13 Neutralization-Reduction Profile Solids Analyses (0 to 37 h)

Run Time h	Sample	Analysis, %							
		As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn
13	Feed	0.42	0.09	7.24	0.68	12.1	16.3	4.54	10.7
	NR1	0.33	0.12	0.62	1.38	12.4	8.74	1.22	35.2
	NR2	0.37	0.09	5.75	0.80	12.4	13.3	3.71	17.6
	NR3	0.41	0.08	5.64	0.78	11.5	13.6	2.96	18.0
	NR4	0.36	0.11	3.16	0.89	11.1	12.1	2.64	24.9
	NR5	0.39	0.11	3.79	0.84	11.1	12.9	2.98	24.5
19	Feed	0.45	0.10	5.71	0.74	13.1	17.9	5.25	11.5
	NR1	0.39	0.10	5.73	0.80	12.3	13.2	4.33	16.8
	NR2	0.41	0.10	5.83	0.78	12.4	12.9	4.52	16.7
	NR3	0.38	0.11	3.04	1.04	12.3	9.9	2.70	28.0
	NR4	0.32	0.07	8.55	0.63	11.8	12.8	4.67	11.5
	NR5	0.35	0.10	5.15	0.81	11.1	11.3	2.98	22.5
25	Feed	0.44	0.09	7.26	0.68	12.0	16.4	4.71	10.6
	NR1	0.42	0.09	6.16	0.78	12.0	12.8	4.18	16.8
	NR2	0.38	0.10	5.15	0.86	11.8	10.9	3.36	22.3
	NR3	0.42	0.09	7.17	0.71	12.1	13.1	4.82	12.6
	NR4	0.38	0.08	8.52	0.66	12.8	12.1	5.61	10.6
	NR5	0.38	0.10	4.43	0.86	11.2	10.5	2.70	25.4
31	Feed	0.46	0.09	7.30	0.67	11.6	17.4	4.74	10.3
	NR1	0.44	0.11	4.78	0.82	11.8	13.0	3.79	20.5
	NR2	0.40	0.09	7.33	0.69	12.3	13.8	5.27	12.1
	NR3	0.37	0.10	4.02	0.86	11.4	10.3	3.04	25.3
	NR4	0.41	0.09	6.93	0.72	12.2	12.1	4.69	15.2
	NR5	0.38	0.10	5.35	0.78	11.2	11.0	3.64	21.3
37	Feed	0.45	0.09	7.57	0.64	10.8	17.5	5.34	9.9
	NR1	0.44	0.09	7.27	0.70	11.8	14.1	5.21	13.1
	NR2	0.42	0.10	6.52	0.68	11.2	13.4	4.65	15.7
	NR3	0.37	0.10	6.11	0.79	13.1	10.6	5.49	17.4
	NR4	0.39	0.08	7.57	0.70	13.1	12.0	6.13	11.7
	NR5	0.51	0.10	6.57	0.75	11.6	12.2	4.52	17.0

Table 8.14 Neutralization-Reduction Profile Solids Analyses (38 to 55 h)

Run Time h	Sample	Analysis, %							
		As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn
43	Feed	0.47	0.10	7.50	0.64	10.0	18.6	5.68	9.6
	NR1	0.44	0.09	7.68	0.71	11.3	14.1	6.19	12.2
	NR2	0.47	0.09	7.69	0.67	11.6	14.6	6.21	11.5
	NR3	0.42	0.09	6.53	0.76	13.0	12.0	6.00	15.0
	NR4	0.47	0.08	8.22	0.68	13.2	12.9	6.69	9.8
	NR5	0.48	0.10	5.88	0.77	11.2	12.5	4.09	19.1
49	Feed	0.43	0.09	7.46	0.65	9.9	18.1	5.61	9.4
	NR1	0.42	0.08	7.22	0.69	10.8	15.0	5.74	11.8
	NR2	0.41	0.08	7.35	0.68	11.5	14.4	6.84	10.8
	NR3	0.42	0.09	7.05	0.72	12.6	12.3	7.16	12.1
	NR4	0.45	0.08	8.16	0.66	12.2	13.1	6.90	9.9
	NR5	0.51	0.09	6.28	0.72	10.7	13.5	4.35	16.5
55	Feed	0.57	0.09	8.26	0.65	9.7	21.6	5.61	8.1
	NR1	0.47	0.08	8.07	0.66	10.0	18.0	6.24	9.5
	NR2	0.44	0.08	8.29	0.68	10.9	17.1	6.86	7.8
	NR3	0.38	0.08	7.61	0.76	13.5	14.2	8.79	8.7
	NR4	0.64	0.10	8.71	0.71	13.3	16.6	8.25	9.8
	NR5	0.72	0.10	8.84	0.68	11.9	17.4	6.96	11.9

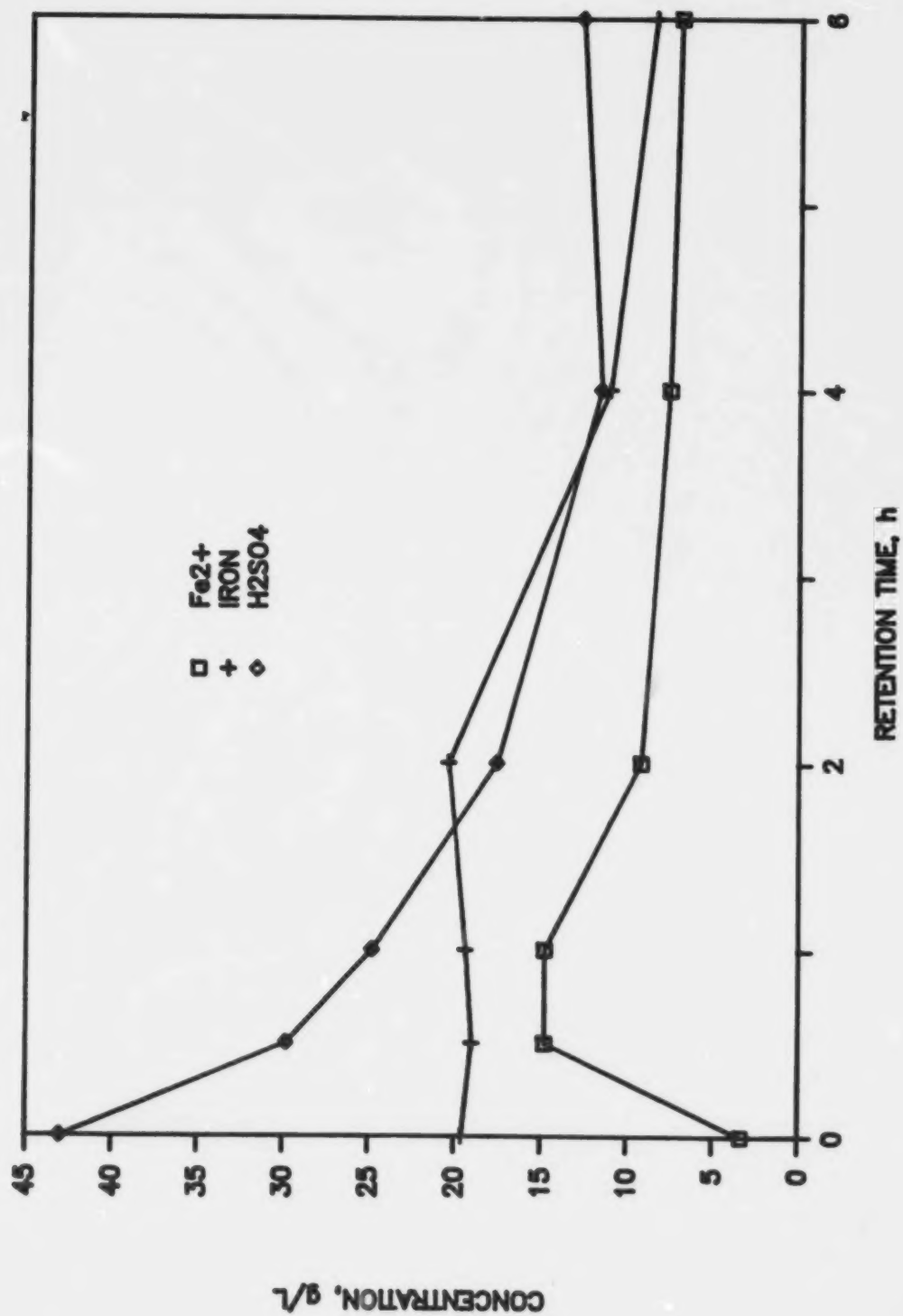


Figure 8.1 Solution Composition in Batch Test 1

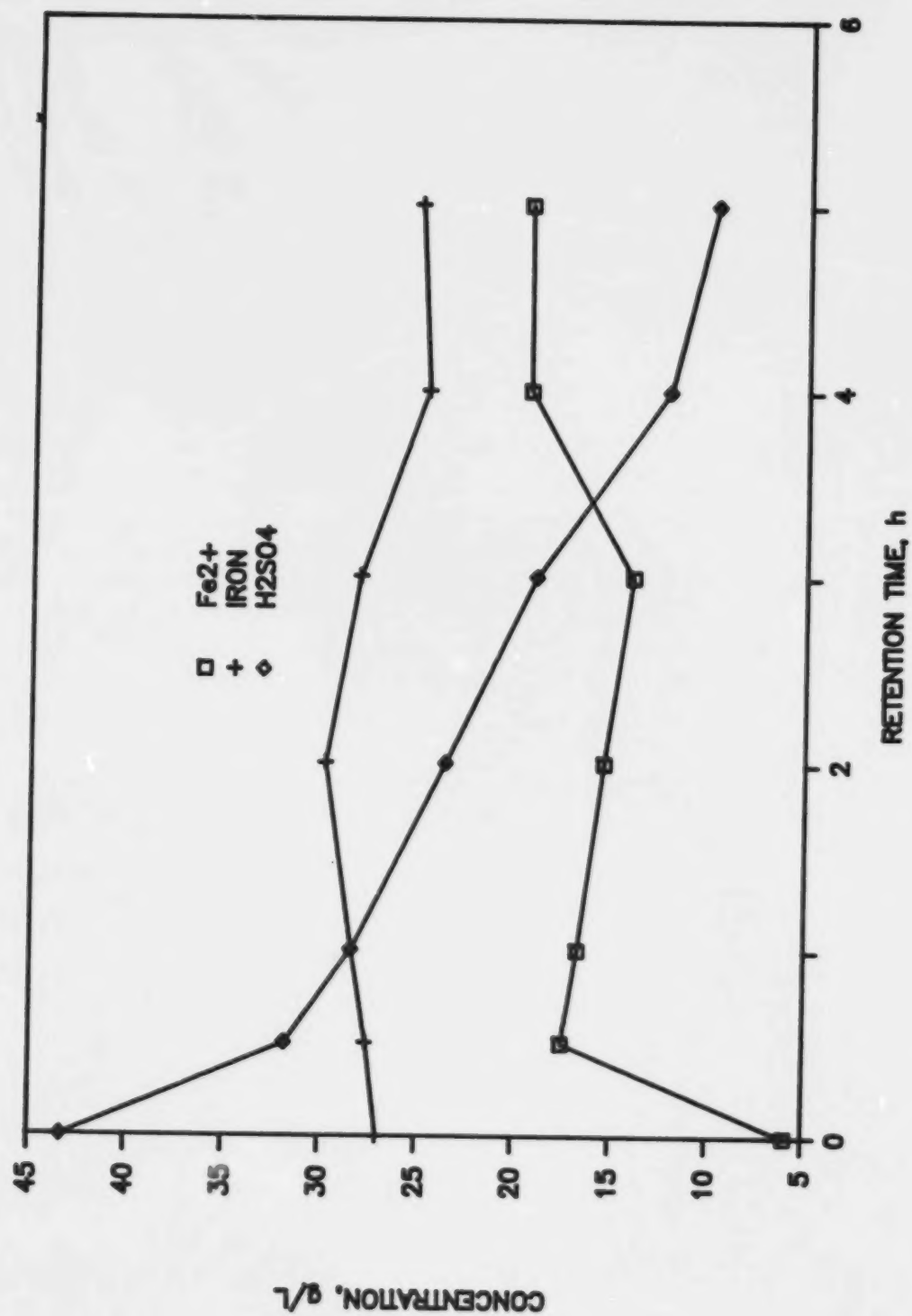


Figure 8.2 Solution Composition in Batch Test 6

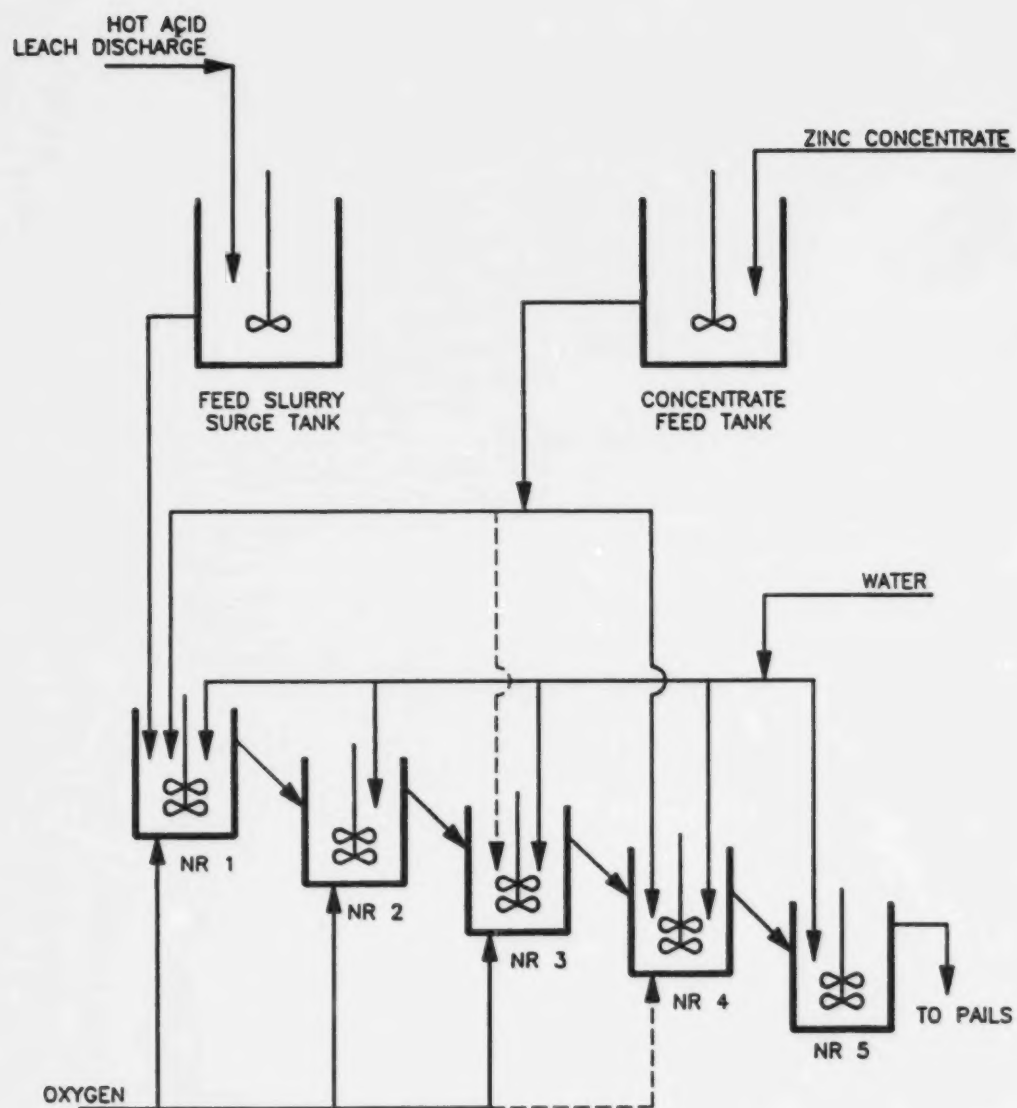


Figure 8.3 Continuous Neutralization-Reduction Equipment Flowsheet

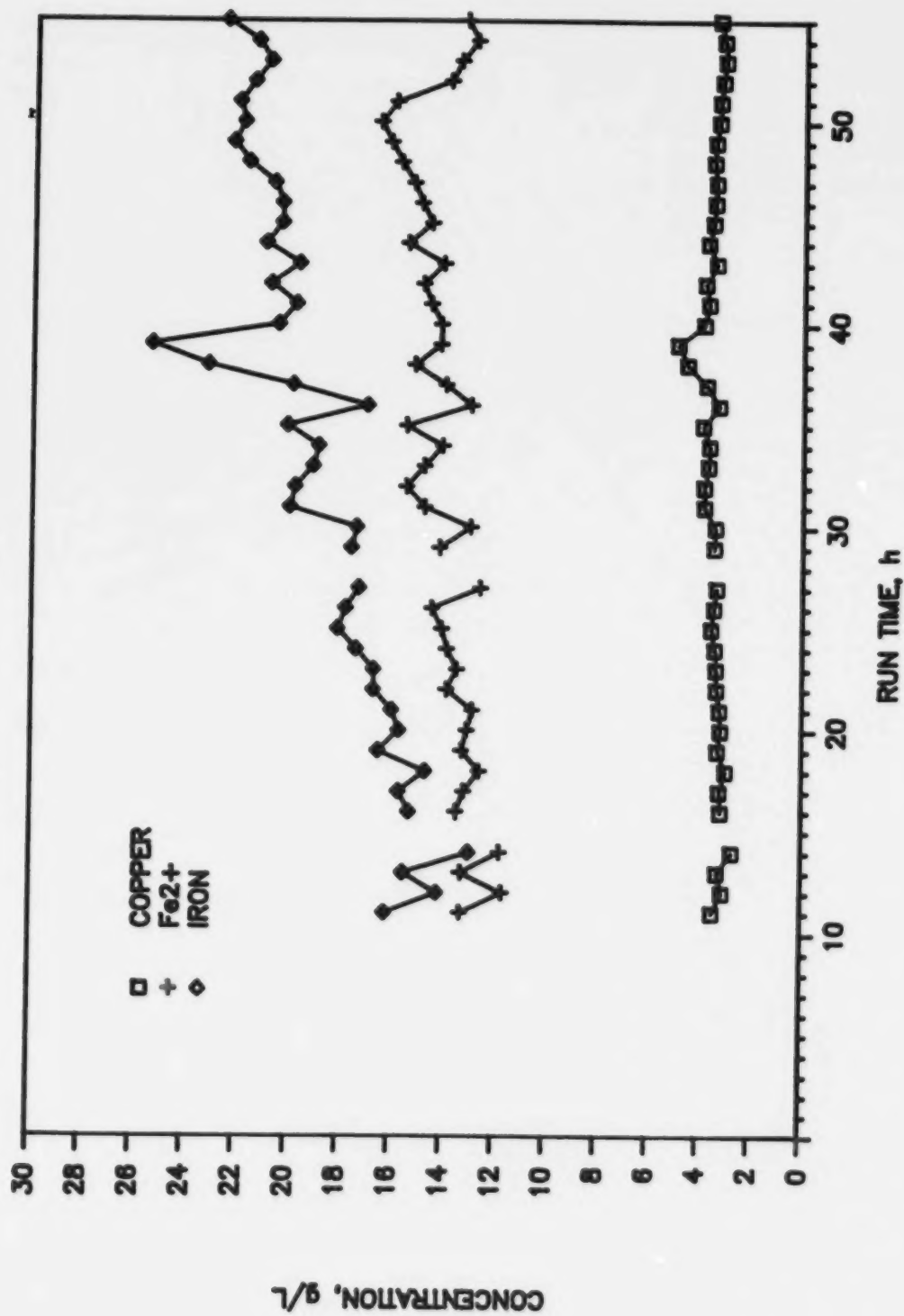


Figure 8.4 Copper and Iron In NR5 Solution

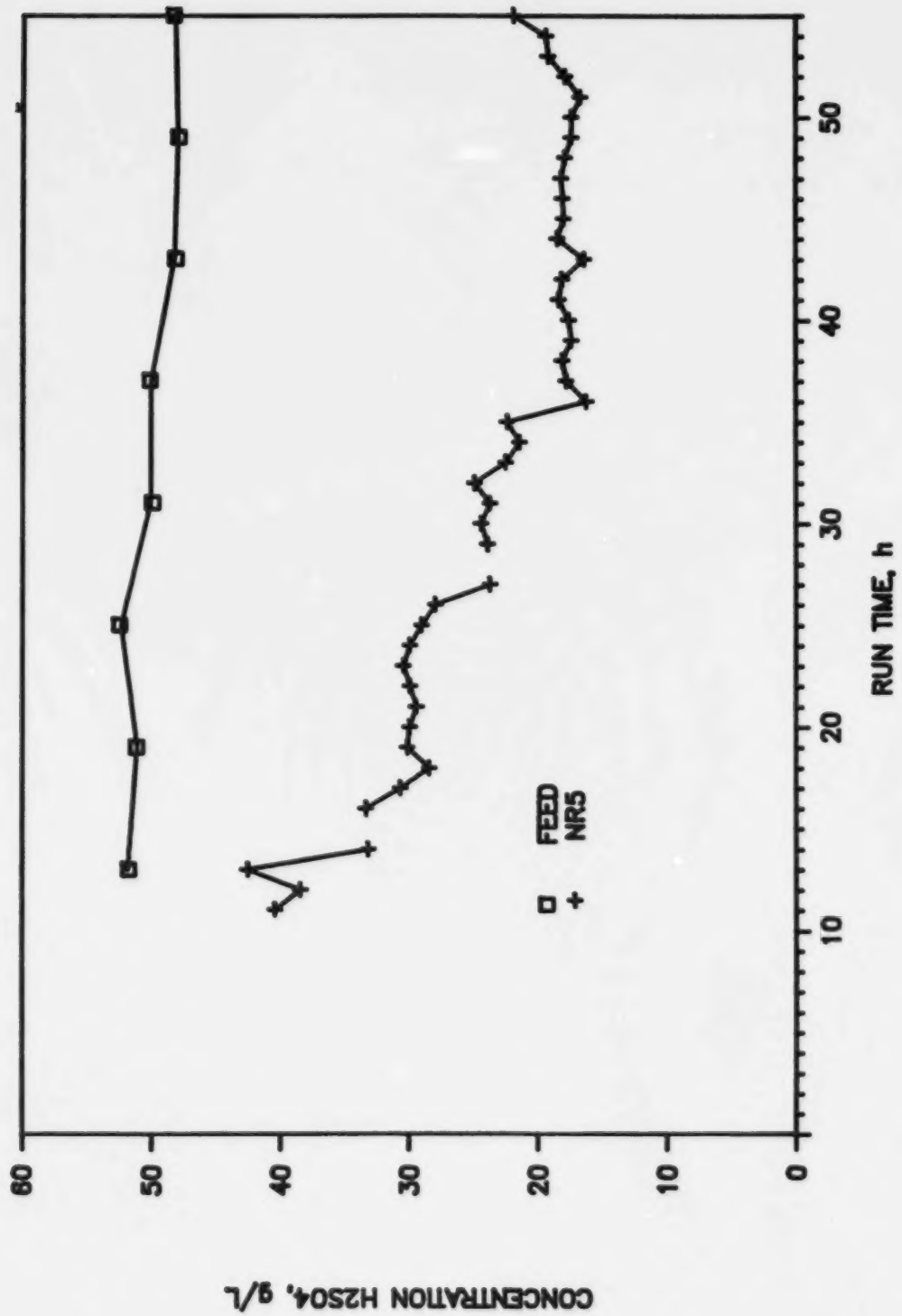


Figure 8.5 Acid In Feed and NR5 Solutions

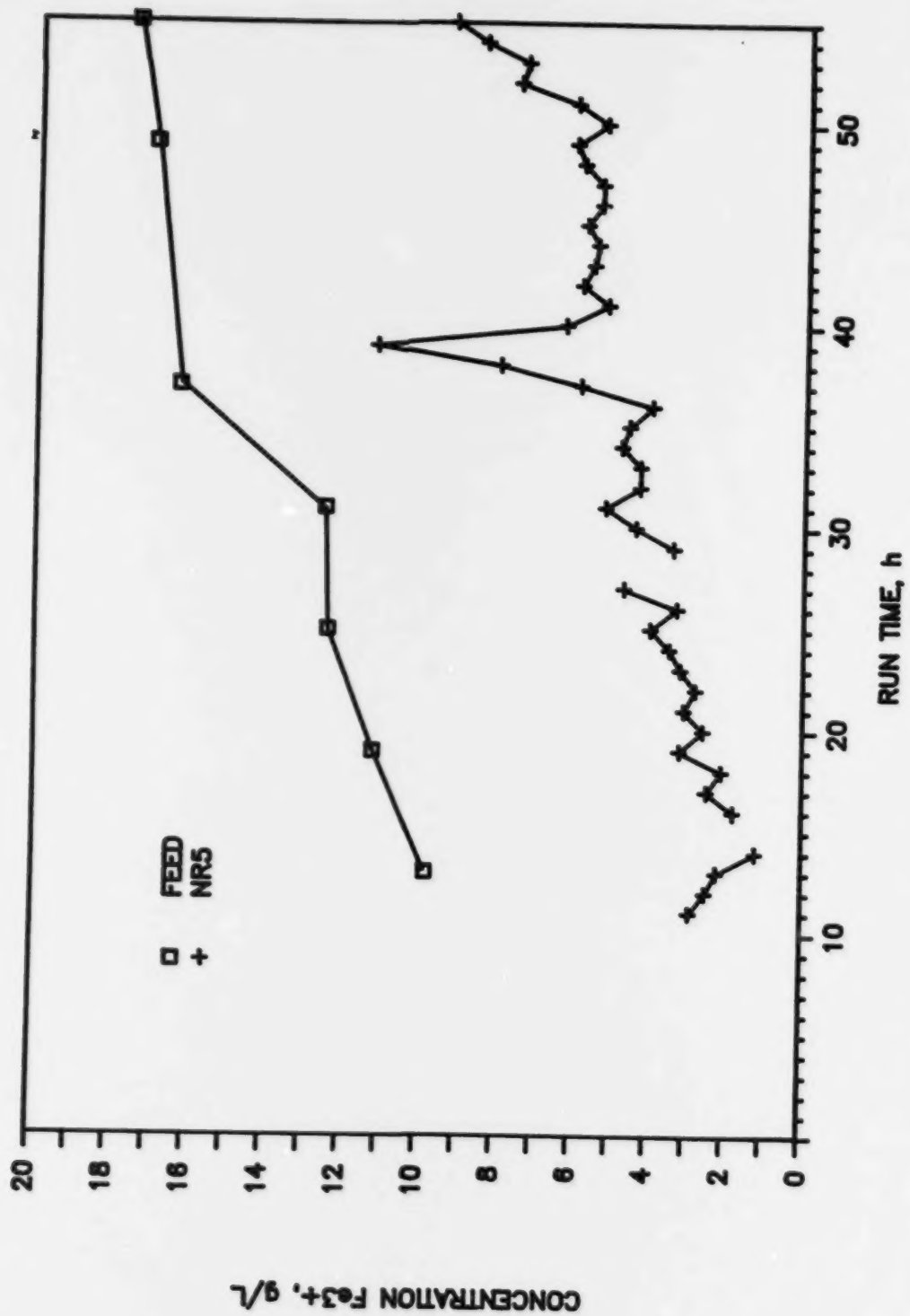


Figure 8.6 Ferric Iron in Feed and NR5 Solutions

9.0 ZINC FLOTATION

Slurry from the neutralization-reduction step was treated directly in the zinc flotation step for recovery of zinc sulphide as a flotation concentrate. This concentrate consisted of excess zinc concentrate added during neutralization-reduction, elemental sulphur formed during neutralization-reduction, and sulphide minerals present in the oxide residue feed that survived the hot acid leach step (principally sphalerite). The zinc flotation tailing slurry was the feed to the reducing leach step.

9.1 BATCH TESTS

Four batch zinc flotation tests were conducted with slurry produced in the neutralization-reduction batch tests. Six tests were performed prior to the continuous zinc flotation test with the continuous neutralization-reduction discharge as the feed. Three additional tests that were performed after completion of the continuous copper flotation test are described in Section 9.3.

9.1.1 Flotation of Batch Neutralization-Reduction Discharge

The first three zinc flotation batch tests were conducted before the continuous testwork. The test conditions and results are given in Tables 9.1 to 9.3. All of the tests were carried out at 75°C. The solids concentration in the rougher-scavenger tests ranged from 6 to 9% by volume. The highest solids concentration (Table 9.1) represented treatment of undiluted neutralization-reduction discharge slurry. Aero 350, a xanthate collector manufactured by Cyanamid, was added to assist flotation, but it is unlikely that this reagent had any effect since the solutions contained at least 10 g/L H₂SO₄. Copper sulphate addition to activate zinc sulphide was unnecessary since the solutions contained about 5 g/L Cu. In any event, the zinc sulphide is probably coated with elemental sulphur, from reaction with ferric iron and sulphuric acid in the neutralization-reduction step, which assists in the flotation.

Rougher-scavenger and cleaner flotation were carried out in Tests 1 and 2, respectively, using neutralization-reduction slurry generated in the treatment of the first sample of oxide residue. The analyses and distributions of the major metals are summarized below and overleaf.

Fraction	Analysis, %			
	Cu	Fe	Pb	Zn
Cleaner Concentrate	0.2	7.1	2.0	37.7
Rougher-Scavenger Concentrate	0.4	9.3	8.1	27.0
Rougher-Scavenger Tailing	0.8	11.6	19.1	4.9

Fraction	Distribution, %			
	Cu	Fe	Pb	Zn
Cleaner Concentrate	4	9	2	51
Cleaner Tailing	7	5	6	3
Rougher-Scavenger Tailing	89	86	92	46

Zinc in the neutralization-reduction solids was approximately equally divided between the cleaner concentrate and the combined cleaner and rougher-scavenger tailing. The zinc in the tailing was primarily oxidic, and was partially extracted in the reducing leach step.

Lead rejection was very efficient, with only 2% reporting to the cleaner concentrate.

The cleaner concentrate also contained 4% of the copper in the neutralization-reduction solids. Much of this copper probably originates from the sulphide concentrate added during neutralization-reduction. About 75% of the copper in the feed materials is in solution at this stage, and the copper in the zinc flotation concentrate represents only about 1% of the total.

The cleaner concentrate contained 37.7% Zn, which is low grade owing to dilution with elemental sulphur produced in the neutralization-reduction step.

Neutralization-reduction slurry generated from treatment of the second sample of oxide residue was used as feed for Test 3 (Table 9.3). The flotation results were similar to those for treatment of the first oxide residue sample.

9.1.2 Flotation of Batch Hot Acid Leach Discharge

A sample of hot acid leach slurry was also subjected to flotation, to simulate a process without the neutralization-reduction step and to provide a measure of the quantity of zinc in the zinc flotation concentrate that originates from the oxide residue feed. The results are presented in Table 9.4 and are summarized overleaf.

The flotation concentrate contained 41% by weight zinc, and 26% of the zinc in the hot acid leach discharge solids. The chemical composition of the concentrate is in agreement with the mineralogical analysis of the oxide residue, which established the presence of sphalerite (See Section 4.1). The results confirm that a significant proportion of the sphalerite in the oxide residue feed material survives the hot acid leach, and is recovered with the zinc flotation concentrate.

Fraction	Analysis, %			
	Cu	Fe	Pb	Zn
Rougher-Scavenger Concentrate	0.6	4.0	10.7	41.4
Rougher-Scavenger Tailing	0.5	9.7	18.3	7.3

Fraction	Distribution, %			
	Cu	Fe	Pb	Zn
Rougher-Scavenger Concentrate	8	2	3	26
Rougher-Scavenger Tailing	92	98	97	74

9.1.3 Flotation of Continuous Neutralization-Reduction Discharge

Six batch zinc flotation tests were performed with continuous neutralization-reduction discharge slurry. The results are given in Tables 9.5 to 9.10, and are summarized below.

Test	Solids % Vol.	Reagents	Analysis, %				Zinc Distribution, %	
			First Conc.		Scav. Tail.		Rough.-Scav. Concentrate	Scavenger Tailing
			Pb	Zn	Pb	Zn		
1	7	none	5.6	36.1	14.3	6.0	81.3	18.7
2	8	Aero 350	5.8	35.9	12.6	7.6	74.2	25.8
3	7	none	n.a.	n.a.	15.4	6.3	79.3	20.7
5	4	none	<0.1	41.8	18.0	6.5	70.4	29.6
6	3	none	2.9	42.1	20.0	6.0	75.4	24.6

n.a. = not analyzed

Tests 1 to 3 were rougher-scavenger tests and were carried out with undiluted slurry. About 80% of the zinc in the feed solids reported to the rougher-scavenger concentrate. The tailing samples contained 6 to 7% Zn and were similar in composition to the tailing obtained for flotation of the hot acid leach slurry (see Section 9.1.2), indicating efficient recovery of the excess zinc concentrate added during neutralization-reduction.

The first cut of rougher-scavenger concentrate in Tests 1 and 2 contained about 36% Zn. A lower pulp density was used in Tests 5 and 6, and the first cut of rougher-scavenger concentrate contained about 42% Zn. Cleaning of the Test 3 rougher concentrate also resulted in a concentrate containing about 42% Zn (Test 4, Table 9.8).

Test 5 was a rougher-scavenger rate test with a combination of fresh feed and scavenger concentrate and cleaner tailing slurries from previous tests, and was more representative of continuous operation than the previous tests. The first cut of concentrate

contained 42% Zn, while the scavenger tailing contained 6.5% Zn. About 30% of the zinc reported to the tailing in this test.

9.2 CONTINUOUS ZINC FLOTATION

Slurry from the continuous neutralization-reduction test was processed through the continuous zinc flotation circuit. The objective was to confirm the results of the batch tests and to generate feed (zinc flotation tailing) for the reducing leach circuit. The products of the continuous flotation were 139 L of cleaner concentrate slurry, containing 65 kg of solids, and 400 L of scavenger tailing slurry, containing 92 kg solids.

9.2.1 Feed Materials

The neutralization-reduction slurry was collected in 20 L pails during continuous demonstration of the neutralization-reduction process. The composition of this slurry was somewhat variable. To provide a homogeneous feed to zinc flotation, a 200 L feed tank was used and pails from the beginning and the end of the neutralization-reduction run were added alternately to the tank. The average composition of the feed slurry, which contained 352 g/L solids on average (8% by volume), is given below. As solution analyses were not obtained during flotation, the analyses given are of the neutralization-reduction discharge solution.

Component	Analysis						
	Cd	Cu	Fe ²⁺	Fe	Pb	H ₂ SO ₄	Zn
Solution, g/L	0.17	3.51	14.1	18.9	-	24.1	81.0
Solids, %	0.10	0.82	-	10.4	10.6	-	24.2

Synthetic solution was added to the cleaner cell to represent the return of cleaner concentrate filtrate and wash water. The composition of this solution is given below.

Solution	Analysis, g/L					
	Cd	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Zn
Synthetic filtrate and wash	<0.01	3.94	20.6	21.0	19.7	79.1

9.2.2 Equipment and Procedures

The continuous flotation test circuit is described in detail in Section 6, and an equipment flowsheet is provided in Figure 9.1. Continuous zinc flotation was conducted in a four cell No. 7 Denver "Sub-A" pilot plant unit of about 94 L total working volume. The unit was divided into one rougher, one cleaner and two scavenger cells. Owing to the short duration of the test, cleaner concentrate filtrate was unavailable and a synthetic solution was pumped to the cleaner cell for dilution of the rougher concentrate slurry.

Cleaner concentrate and scavenger tailing slurries were collected in pails. The feed to flotation was exhausted after 4.5 h, and operation of the unit was continued in a batch mode to produce additional feed for the reducing leach.

9.2.3 Operating Parameters

The operating parameters for the continuous flotation test are given in Table 9.11. Samples were collected in an order which least upset the operation. Scavenger tailing, cleaner concentrate, rougher concentrate, scavenger concentrate, feed slurry and feed solution (filtrate and wash) streams were collected in the order stated. The feed rates to the circuit were initially 1.1 L/min of neutralization-reduction slurry to the rougher cell and 0.14 L/min of synthetic filtrate and wash solution to the cleaner cell. These rates were increased to 2.0 L/min and 0.28 L/min, respectively, after 2 h of operation.

9.2.4 Results

The results of the continuous flotation tests are given in Tables 9.12 to 9.14. Average analyses and distributions are given in the tables below.

Fraction	Analysis, %						
	Cu	Fe	Pb	S°	S(SO ₄)	S	Zn
Feed	0.82	10.4	10.6	5.4	6.0	22.3	24.2
Cleaner Concentrate	1.06	9.6	2.8	9.1	0.7	37.2	43.8
Rougher Concentrate	1.02	10.1	6.4	5.1	2.1	31.0	39.1
Scavenger Concentrate	0.96	10.0	10.1	2.9	3.7	25.8	33.1
Scavenger Tailing	0.59	10.8	17.3	3.4	9.9	11.2	7.3

Fraction	Weight kg	Distribution, %					
		Cu	Fe	Pb	S°	S	Zn
Cleaner Concentrate	64.6	55.8	38.4	10.2	65.3	70.0	80.8
Scavenger Tailing	92.0	44.2	61.6	89.8	34.7	30.0	19.2

The cleaner concentrate composition ranged from 43.4 to 44.3% Zn, with an average of 43.8% Zn, which was similar to the results for batch tests with the same feed (see Section 9.1.3). However, the continuous scavenger tailing contained 6.4 to 8.0% Zn (7.3% on average), compared with typical values of 6.0 to 6.5% Zn in the batch tests. The concentration of zinc in the scavenger tailing increased in the second half of the continuous flotation operation, when the feed rate was increased (see Tables 9.11 and 9.13). The residual zinc sulphide in the tailing was collected along with the copper flotation concentrate after the reducing leach step, which reduced the grade of the copper concentrate significantly (see Section 11).

The concentration of lead in the cleaner concentrate was 2.8% on average, which represented 10% of the lead in the feed. A batch recleaner flotation test with the continuous cleaner concentrate as feed (Table 9.15) produced a concentrate containing 1% Pb. A second stage of cleaning, therefore, would increase lead rejection during zinc flotation, and increase lead recovery to the lead concentrate. Optimization of concentrate addition in the neutralization-reduction step would also reduce the quantity of zinc flotation concentrate, and therefore also result in improved recovery of lead to the lead flotation concentrate. With recleaning, the concentrate produced in the continuous zinc flotation test would contain about 4.0% of the lead in the feed and with optimization of concentrate addition in neutralization-reduction, this could be reduced to between 2 and 3%. This is in good agreement with the first batch tests, where 2% of the lead reported to the cleaner concentrate (see Section 9.1.1).

During the continuous test, the scavenger tailing solids averaged 3.4% elemental sulphur, representing nearly 35% of the elemental sulphur in the feed to flotation. Sulphur that did not float may be present as very fine particles. An improved recovery of zinc and elemental sulphur to the zinc flotation concentrate may be achieved by using a coarser zinc concentrate as the feed in the neutralization-reduction step.

The cleaner concentrate produced in the continuous flotation test contained 1.06% Cu on average, which is very close to the composition of the concentrate fed to the neutralization-reduction circuit. Copper is partially extracted in the neutralization-reduction circuit, and there is no net loss of copper from the oxide residue to the zinc flotation concentrate (see Section 8.3.8). In comparison, the concentrate produced by direct flotation of the oxide residue in Phase Ib of the project contained 4.55 Cu, or 9% of the copper in the oxide residue (see Phase Ib report, Section 8).

9.3 ADDITIONAL BATCH TESTS

Scavenger tailing slurry from the continuous zinc flotation test was the feed for the continuous reducing leach test (Section 10). The reducing leach slurry was then treated by flotation to recover a copper concentrate (Section 11). Scavenger flotation in the continuous zinc flotation test was incomplete, however, and the tailing contained 1 to 1.5% Zn as zinc sulphide, as described in Section 11.3. This zinc sulphide was recovered with copper sulphide in the copper flotation step and as copper in the oxide residue is low, the resulting concentrate contained almost as much zinc as copper. Additional tests were therefore carried out to establish that a higher grade copper concentrate could be produced. The additional zinc flotation tests are described in Tables 9.16 to 9.18 (Supplementary Tests 8 to 10).

The feed material for Tests 8 to 10 was the last pail of discharge slurry produced in the continuous neutralization-reduction test. The same feed was used for the earlier batch tests, as described in Tables 9.5 to 9.10.

The collector Z200, manufactured by Dow, was added to assist zinc sulphide flotation in Tests 8 to 10, but appeared to have little effect as the concentration of zinc in the tailing was similar to earlier batch tests without Z200. In Test 8, Z200 was added near the end of flotation, and there was virtually no difference in the zinc concentration of the tailing after reagent addition (Table 9.16).

Tailing solids collected in Tests 8 and 9 contained about 6% Zn, which is significantly lower than that produced in the continuous zinc flotation tests (7 to 8% Zn). The combined batch tailing was used as feed for batch reducing leach and copper flotation tests (see Section 11).

Tests 10 was a cleaner test, using a rougher concentrate produced under the same conditions as in Test 9. Lead was successfully rejected, with the first two cuts of cleaner concentrate containing about 44% Zn and only 1 to 2% Pb. This test confirmed that addition of Z200 in zinc flotation would not have a deleterious effect on eventual lead recovery. It would appear, however, that reagents are not required in the zinc flotation step.

9.4 CONCLUSIONS AND RECOMMENDATIONS

Zinc sulphide and elemental sulphur may be conveniently recovered from the neutralization-reduction discharge slurry by flotation. Flotation reagents are not required for recovery of zinc, since zinc sulphide is in intimate contact with elemental sulphur in the feed solids. The zinc flotation concentrate can be fed to a zinc pressure leach circuit for recovery of the zinc and sulphur values.

Careful attention to the scavenger flotation operation is required to minimize the zinc content of the scavenger tailing and to permit production of a high grade copper concentrate in the copper flotation step.

Table 9.1 Batch Zinc Flotation: Test 1

Feed Material	: Neutralization-reduction discharge slurry (Blend of Tests 1 to 3)
Solids Concentration	: 335 g/L slurry (9% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (10 g/L)
Temperature	: 75°C
Reagents	: 75 g/t Aero 350
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (60 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	1	2	5	-
Weight, g	669.0	72.1	25.2	18.0	553.7
Analysis, %					
As	2.94	1.37	1.59	2.99	3.20
Cd	0.08	0.14	0.15	0.12	0.07
Ca	5.28	1.03	0.67	1.61	6.16
Cu	0.76	0.41	0.43	0.77	0.82
Fe	11.2	8.67	8.98	10.7	11.6
Pb	17.2	6.95	7.73	14.1	19.1
SiO ₂	3.52	3.28	5.16	5.88	3.40
Zn	8.7	28.7	29.7	16.4	4.89
Distribution, %					
Cu	100	5.8	2.1	2.7	89.3
Fe	100	8.4	3.0	2.6	86.0
Pb	100	4.3	1.7	2.2	91.8
Zn	100	35.6	12.9	5.1	46.5

Table 9.2 Batch Zinc Flotation: Test 2 (Cleaner)

Feed Material	: Rougher-scavenger concentrate resulting from 5 minutes of flotation of neutralization-reduction discharge under conditions of Test 1.
Solids Concentration	: 50 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (10 g/L)
Temperature	: 75°C
Reagents	: 500 g/t Aero 350
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (400 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	1	2	5	-
Weight, g	99.9	63.7	11.0	3.9	21.3
Analysis, %					
As	1.47	0.48	0.41	0.59	5.15
Cd	0.16	0.18	0.17	0.16	0.10
Ca	0.06	0.06	0.02	<0.005	0.07
Cu	0.42	0.21	0.20	0.24	1.22
Fe	8.97	7.09	6.96	7.45	15.9
Pb	7.09	2.00	1.94	3.05	25.7
SiO ₂	3.31	2.77	3.65	5.65	4.34
Zn	31.2	38.1	36.8	34.4	7.21
Distribution, %					
Cu	100	31.5	5.2	2.2	61.1
Fe	100	50.4	8.5	3.2	37.8
Pb	100	18.0	3.0	1.7	77.3
Zn	100	77.8	13.0	4.3	4.9

Table 9.3 Batch Zinc Flotation: Test 3

Feed Material	: 1.24 L neutralization-reduction discharge slurry (Test 4) diluted to 2.0 L with 10 g/L H ₂ SO ₄ solution.
Solids Concentration	: 234 g/L slurry (6% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (10 g/L)
Temperature	: 75°C
Reagents	: 50 g/t Aero 350
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (85 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	1	2	5	-
Weight, g	467.2	45.4	16.5	14.2	391.1
Analysis, %					
As	0.83	0.50	0.50	0.79	0.88
Cd	0.08	0.16	0.16	0.14	0.07
Ca	6.73	0.71	0.10	0.36	7.94
Cu	0.42	0.32	0.33	0.37	0.44
Fe	12.2	6.72	6.91	9.53	13.1
Pb	16.0	6.71	6.83	13.4	17.5
SiO ₂	4.76	4.74	5.28	7.46	4.64
Zn	9.01	25.5	25.5	20.1	6.07
Distribution, %					
Cu	100	7.4	2.8	2.7	87.2
Fe	100	5.4	2.0	2.4	90.2
Pb	100	4.1	1.5	2.6	91.8
Zn	100	27.3	9.9	6.7	56.0

Table 9.4 Batch Zinc Flotation: Test 4 (Hot Acid Leach Slurry)

Feed Material	: 1.30 L hot acid leach slurry (Test 4) diluted to 2.0 L with 50 g/L H ₂ SO ₄ solution.
Solids Concentration	: 232 g/L slurry (6% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (50 g/L)
Temperature	: 75°C
Reagents	: 100 g/t Aero 350
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (85 g/t)

Solids	Calc Head	Con 1	Tailing
Time, min	-	3	-
Weight, g	464.8	26.8	438.0
Analysis, %			
As	0.44	0.28	0.45
Cd	0.09	0.30	0.08
Ca	7.66	1.16	8.06
Cu	0.48	0.63	0.47
Fe	9.37	3.95	9.70
Pb	17.9	10.7	18.3
SiO ₂	5.99	1.69	6.25
Zn	9.23	41.4	7.26
Distribution, %			
Cu	100	8	92
Fe	100	2	98
Pb	100	3	97
Zn	100	26	74

Table 9.5 Batch Zinc Flotation: Supplementary Test 1

Feed Material	: Continuous neutralization-reduction discharge slurry, collected between 53 and 55 h run time
Solids Concentration	: 275 g/L slurry (7% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: none
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	4	8	-
Weight, g	546.9	108.7	44.0	38.5	86.1	269.6
Analysis, %						
As	0.41	0.20	0.30	0.53	0.58	0.44
Cd	0.09	0.12	0.14	0.13	0.08	0.07
Ca	6.70	0.95	0.79	4.76	8.23	9.79
Cu	0.71	0.90	0.99	0.80	0.64	0.59
Fe	10.3	9.07	9.43	9.96	10.1	11.1
Pb	12.9	5.55	6.93	16.2	19.2	14.3
SiO ₂	4.35	1.39	2.12	4.33	4.97	5.72
S°	5.2	11.8	9.0	2.8	1.7	3.5
Zn	15.9	36.1	37.2	20.0	8.64	6.02
Distribution, %						
Cu	100	25.3	11.3	8.0	14.3	41.2
Fe	100	17.5	7.4	6.8	15.4	53.0
Pb	100	8.6	4.3	8.9	23.5	54.8
Zn	100	45.1	18.8	8.8	8.6	18.7

Table 9.6 Batch Zinc Flotation: Supplementary Test 2

Feed Material	: Continuous neutralization-reduction discharge slurry, collected between 53 and 55 h run time
Solids Concentration	: 312 g/L slurry (8% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: Aero 350, 50 g/t
Conditioning Time	: 5 minutes
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	4	8	-
Weight, g	624.6	94.4	72.9	68.5	52.6	336.2
Analysis, %						
As	0.42	0.24	0.28	0.55	0.67	0.44
Cd	0.09	0.12	0.14	0.11	0.08	0.07
Ca	6.25	1.03	1.25	5.81	7.12	8.75
Cu	0.71	0.96	0.93	0.69	0.63	0.61
Fe	10.1	9.92	9.36	9.03	9.16	10.7
Pb	12.1	5.77	7.48	17.0	20.4	12.6
SiO ₂	4.35	1.35	1.86	4.65	6.30	5.36
S°	4.9	11.9	9.4	3.5	2.8	2.6
Zn	15.9	35.9	35.3	15.3	6.51	7.60
Distribution, %						
Cu	100	20.4	15.3	10.6	7.5	46.2
Fe	100	14.8	10.8	9.8	7.6	57.0
Pb	100	7.2	7.2	15.4	14.2	56.0
Zn	100	34.2	26.0	10.6	3.5	25.8

Table 9.7 Batch Zinc Flotation: Supplementary Test 3

Feed Material	: Continuous neutralization-reduction discharge slurry, collected between 53 and 55 h run time
Solids Concentration	: 290 g/L slurry (7% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: none
Frothing Agent	: none

Solids	Head	Con 1*	Con 2**	Tailing
Time, min	-	3	5	-
Weight, g	580	n.a.	n.a.	306
Analysis, %				
As	0.42	n.a.	n.a.	0.53
Cd	0.09	n.a.	n.a.	0.07
Ca	6.48	n.a.	n.a.	10.1
Cu	0.71	n.a.	n.a.	0.60
Fe	10.2	n.a.	n.a.	11.1
Pb	12.5	n.a.	n.a.	15.4
SiO ₂	4.53	n.a.	n.a.	6.17
Zn	15.9	n.a.	n.a.	6.29

* Rougher ** Scavenger
n.a. = not analyzed

Table 9.8 Batch Zinc Flotation: Supplementary Test 4

Feed Material	: Rougher concentrate slurry from supplementary Test 3, diluted with filtrate to 1.0 L; 0.3 L filtrate added during flotation
Slurry Volume	: 1.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 25°C
Reagents	: none
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	4	8	-
Weight, g	n.a.	53	38	27	9	n.a.
Analysis, %						
As	n.a.	0.10	0.16	0.15	0.44	n.a.
Cd	n.a.	0.02	0.04	0.07	0.08	n.a.
Ca	n.a.	0.32	0.33	0.31	0.79	n.a.
Cu	n.a.	0.59	0.65	0.68	0.60	n.a.
Fe	n.a.	8.84	9.23	9.16	9.63	n.a.
Pb	n.a.	<0.05	<0.05	<0.05	11.6	n.a.
SiO ₂	n.a.	1.01	1.20	1.29	4.18	n.a.
S°	n.a.	14.7	12.7	10.3	5.4	n.a.
Zn	n.a.	41.6	44.4	45.6	34.6	n.a.

n.a. = not analyzed

Table 9.9 Batch Zinc Flotation: Supplementary Test 5

Feed Material	: Scavenger concentrate slurry from Supplementary Test 3; cleaner tailing slurry from Supplementary Test 4; 0.78 L continuous neutralization-reduction discharge collected between 53 and 55 h run time.
Solids Concentration	: 144 g/L slurry (4% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: none
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	4	8	-
Weight, g	288	51	13	14	15	195
Analysis, %						
As	0.57	0.21	0.41	0.72	0.86	0.64
Cd	0.08	0.05	0.10	0.15	0.10	0.08
Ca	6.42	0.50	0.82	2.29	3.87	8.84
Cu	0.64	0.64	0.56	0.78	0.74	0.63
Fe	10.3	9.35	9.59	9.75	10.1	10.6
Pb	14.9	<0.05	8.50	19.6	25.6	18.0
SiO ₂	5.55	1.59	3.71	6.21	5.89	6.64
S°	3.50	12.5	5.1	1.8	1.7	1.3
Zn	14.9	41.8	37.6	19.3	9.42	6.53
Distribution, %						
Cu	100	17.7	3.9	5.9	6.0	66.5
Fe	100	16.1	4.2	4.6	5.1	69.9
Pb	100	<0.1	2.6	6.4	9.0	82.0
Zn	100	49.5	11.4	6.3	3.3	29.6

Table 9.10 Batch Zinc Flotation: Supplementary Test 6

Feed Material	: Continuous neutralization-reduction discharge slurry, collected between 53 and 55 h run time, diluted with filtrate
Solids Concentration	: 132 g/L slurry (3% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: none
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	4	8	-
Weight, g	264	64	9	8	13	170
Analysis, %						
As	0.44	0.19	0.46	0.76	0.84	0.49
Cd	0.05	0.14	0.18	0.10	0.08	<0.01
Ca	6.57	0.08	1.02	3.44	5.09	9.56
Cu	0.47	1.01	0.87	0.75	0.68	0.21
Fe	10.3	9.30	9.71	10.1	9.88	10.7
Pb	15.9	2.86	12.7	24.1	24.2	20.0
SiO ₂	4.89	0.81	4.33	7.63	8.19	6.08
S°	3.52	12.1	4.0	1.4	1.7	0.5
Zn	15.8	42.1	31.1	10.5	6.40	6.03
Distribution, %						
Cu	100	52.6	6.4	4.9	7.2	29.0
Fe	100	22.0	3.2	3.0	4.7	67.1
Pb	100	4.4	2.7	4.6	7.5	80.9
Zn	100	64.7	6.7	2.0	2.0	24.6

Table 9.11 Zinc Flotation Continuous Operating Parameters

Run Time h	Sample	Rate mL/min	Pulp Density g/L	Solids*		Retention Time	
				Wt %	Vol %	Cell	min
1	Feed Slurry	1140	1462	25	8		
	Cleaner Con.	414	1570	29	11	Cleaner	9.9
	Rougher Con.	2282	1443	16	6	Rougher	3.0
	Scav. Con.	4980	1406	14	5	Scav.	8.2
	Scav. Tailing	220	1446	15	5		
	Filtrate Return	140	1258	-	-		
2	Feed Slurry	1160	1497	22	8		
	Cleaner Con.	317	1585	30	12	Cleaner	19.9
	Rougher Con.	1068	1535	24	9	Rougher	4.7
	Scav. Con.	3014	1422	15	5	Scav.	12.0
	Scav. Tailings	870	1428	15	5		
	Filtrate Return	138	1270	-	-		
3	Feed Slurry	1992	1474	23	8		
	Cleaner Con.	496	1605	31	12	Cleaner	19.0
	Rougher Con.	986	1639	26	10	Rougher	5.3
	Scav. Con.	1740	1504	23	9	Scav.	13.6
	Scav. Tailing	1770	1448	16	6		
	Filtrate Return	280	1283	-	-		
4	Feed Slurry	1980	1492	25	9		
	Cleaner Con.	462	1600	31	12	Cleaner	18.8
	Rougher Con.	996	1601	30	12	Rougher	5.0
	Scav. Con.	2000	1533	24	9	Scav.	12.6
	Scav. Tailing	1580	1462	17	6		
	Filtrate Return	281	n.a.	-	-		

* Based on weights of dry solids collected.

n.a. = not analyzed

Table 9.12 Continuous Zinc Flotation Analyses (As to Fe)

Profile	Stream	Analysis, %				
		As	Cd	Ca	Cu	Fe
1 (1 h)	Feed	0.36	0.10	4.47	0.82	10.2
	Cleaner Con.	0.17	0.13	<0.005	1.06	9.44
	Rougher Con.	0.40	0.12	2.85	0.88	10.1
	Scavenger Con.	0.51	0.10	4.30	0.78	10.1
	Scavenger Tailing	0.56	0.07	9.06	0.56	10.6
2 (2 h)	Feed	0.34	0.10	4.51	0.83	10.4
	Cleaner Con.	0.20	0.13	<0.005	1.06	9.51
	Rougher Con.	0.28	0.14	0.76	1.03	10.0
	Scavenger Con.	0.53	0.12	3.14	0.87	10.3
	Scavenger Tailing	0.58	0.07	8.86	0.58	10.8
3 (3 h)	Feed	0.42	0.10	4.62	0.82	10.7
	Cleaner Con.	0.22	0.13	<0.005	1.07	9.82
	Rougher Con.	0.19	0.15	<0.005	1.08	10.1
	Scavenger Con.	0.29	0.17	0.14	1.09	9.93
	Scavenger Tailing	0.52	0.07	8.41	0.62	11.1
4 (4 h)	Feed	0.39	0.09	4.77	0.81	9.99
	Cleaner Con.	0.20	0.13	<0.005	1.07	9.82
	Rougher Con.	0.22	0.15	<0.005	1.09	10.2
	Scavenger Con.	0.29	0.17	0.50	1.11	9.83
	Scavenger Tailing	0.50	0.07	8.38	0.59	10.7
Average	Feed	0.38	0.10	4.59	0.82	10.4
	Cleaner Con.	0.20	0.13	<0.005	1.06	9.65
	Rougher Con.	0.27	0.14	0.90	1.02	10.1
	Scavenger Con.	0.40	0.14	2.02	0.96	10.0
	Scavenger Tailing	0.54	0.07	8.68	0.59	10.8

Table 9.13 Continuous Zinc Flotation Analyses (Pb to Zn)

Profile	Stream	Analysis, %					
		Pb	SiO ₂	S°	S(SO ₄)	S	Zn
1 (1 h)	Feed	10.4	3.06	4.3	5.30	22.8	24.9
	Cleaner Con.	2.90	1.26	8.8	0.75	35.1	43.4
	Rougher Con.	11.9	3.15	3.5	4.67	23.6	27.9
	Scavenger Con.	15.1	3.83	2.8	6.64	18.7	20.4
	Scavenger Tailing	17.7	4.66	0.5	9.90	11.0	6.44
2 (2 h)	Feed	10.5	3.15	4.2	5.61	23.2	25.1
	Cleaner Con.	2.78	1.28	9.2	0.66	36.9	43.8
	Rougher Con.	6.58	1.84	4.7	2.01	30.5	39.0
	Scavenger Con.	14.2	3.89	2.2	5.13	21.1	25.0
	Scavenger Tailing	17.6	4.86	4.3	9.77	11.1	6.86
3 (3 h)	Feed	11.3	3.21	6.3	5.96	21.3	24.0
	Cleaner Con.	2.74	1.16	9.3	0.60	38.2	43.8
	Rougher Con.	3.37	1.16	6.3	0.82	35.2	44.9
	Scavenger Con.	5.61	2.12	3.4	1.33	31.7	43.3
	Scavenger Tailing	17.3	4.49	4.4	10.0	31.2	7.99
4 (4 h)	Feed	10.2	2.65	6.6	7.15	21.9	22.8
	Cleaner Con.	2.63	0.98	9.2	0.62	38.5	44.3
	Rougher Con.	3.69	0.79	5.9	0.89	34.9	44.7
	Scavenger Con.	5.47	1.50	3.3	1.68	31.5	43.7
	Scavenger Tailing	16.5	4.60	4.3	9.99	11.4	8.02
Average	Feed	10.6	3.02	5.4	6.00	22.3	24.2
	Cleaner Con.	2.76	1.17	9.1	0.66	37.2	43.8
	Rougher Con.	6.38	1.74	5.1	2.10	31.0	39.1
	Scavenger Con.	10.1	2.84	2.9	3.70	25.8	33.1
	Scavenger Tailing	17.3	4.65	3.4	9.92	11.2	7.33

Table 9.14 Continuous Zinc Flotation Product Inventory

Cleaner Concentrate			Scavenger Tailing*		
Pail No.	Slurry Weight kg	Slurry P.D. g/L	Pail No.	Slurry Weight kg	Slurry P.D. g/L
1 [*]	30.7	1587	1	18.7	1435
2	26.1	1567	2	17.3	1439
3	30.5	1553	3	17.8	
4	29.4	1552	4	17.8	
5	28.9	1638	5	17.2	1439
6	28.4	1594	6	17.6	
7	9.5	1624	7	19.3	
8**	16.2	1624	8	18.0	
9**	17.9	1342	9	17.9	
Total	217.5	kg slurry	10	17.2	1436
	64.6	kg solids	11	18.2	
			12	17.2	
			13	17.5	
			14	17.6	
			15	18.3	1439
			16	17.5	
			17	18.2	
			18	17.1	
			19	17.3	
			20	17.9	1435
			21	15.7	
			22	15.6	
			23	15.1	
			24	15.0	
			25	16.1	1425
			26	18.4	
			27 to 30**	118.5	1384
			Total	570.2	kg slurry
				92.0	kg solids

* Products blended before distributing to pails.

** Collected while operating in batch mode.

Table 9.15 Batch Zinc Flotation: Supplementary Test 7

Feed Material	: Continuous zinc flotation cleaner concentrate (Pail Number 6, 0.6 L) diluted with 1.4 L filtrate
Solids Concentration	: 133 g/L slurry (3% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: none
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	1	2	4	-
Weight, g	266.0	137.5	68.6	34.2	25.7
Analysis, %					
As	0.18	0.14	0.08	0.17	0.69
Cd	0.13	0.13	0.14	0.17	0.10
Ca	<0.005	<0.005	<0.005	<0.005	<0.005
Cu	1.07	1.01	1.07	1.17	1.28
Fe	9.76	9.55	9.57	10.1	10.9
Pb	2.62	1.06	0.71	1.16	18.0
SiO ₂	0.94	0.23	<0.05	0.83	7.29
S	37.6	40.3	37.0	37.1	25.1
Zn	44.1	46.0	47.9	48.1	18.6
Distribution, %					
Cu	100	48.7	25.7	14.0	11.5
Fe	100	50.6	25.3	13.3	10.8
Pb	100	20.9	7.0	5.7	66.4
Zn	100	53.9	28.0	14.0	4.1

Table 9.16 Batch Zinc Flotation: Supplementary Test 8

Feed Material	: Continuous neutralization-reduction slurry, collected between 53 and 55 h run time.
Solids Concentration	: 237 g/L slurry (6% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: Z200, 0.2 g added after 10 minutes
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (40 g/t)

Solids	Calc. Head	Con. 1	Con. 2	Con. 3	Con. 4	Tail 1*	Con. 5	Tail 2
Time, min	-	1	2	5	10	-	15	-
Weight, g	473.3	97.7	13.7	41.2	40.1	21.3	15.7	243.6
Analysis, %								
Cd	0.08	0.11	0.12	0.15	0.11	0.06	0.08	0.06
Ca	5.86	0.87	<0.01	0.14	3.35	9.34	2.03	9.52
Cu	0.68	0.91	1.00	0.94	0.70	0.55	0.72	0.54
Fe	9.88	9.06	9.19	9.79	9.40	10.3	10.1	10.3
Pb	13.3	5.61	3.11	7.89	22.7	16.1	29.7	15.0
SiO ₂	4.93	1.79	1.28	3.02	6.24	6.36	11.3	5.96
S°	5.1	13.1	14.3	7.9	2.8	1.9	1.5	1.8
S(SO ₄)	7.24	2.01	0.70	1.81	7.06	10.4	6.76	10.4
S	18.7	36.3	41.3	32.9	13.9	10.7	8.00	10.1
Zn	16.4	36.0	40.6	36.8	13.8	5.94	6.53	5.79
Distribution, %								
Cu	100	27.4	4.2	12.0	8.7	3.6	3.5	40.6
Fe	100	18.9	2.7	8.6	8.1	4.7	3.4	53.6
Pb	100	8.7	0.7	5.2	14.5	5.4	7.4	58.1
Zn	100	45.2	7.1	19.5	7.1	1.6	1.3	18.1

* Sampled before addition of Z200.

Table 9.17 Batch Zinc Flotation: Supplementary Test 9

Feed Material	: Continuous neutralization-reduction slurry, collected between 53 and 55 h run time
Solids Concentration	: 241 g/L slurry (6% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: Z200, 420 g/t
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (40 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	5	10	-
Weight, g	482.0	99.0	35.2	56.9	43.6	247.3
Analysis, %						
Cd	0.09	0.11	0.12	0.12	0.10	0.07
Ca	5.43	2.33	0.84	1.63	3.68	8.51
Cu	0.70	0.82	0.90	0.86	0.74	0.58
Fe	9.98	9.06	9.37	9.34	9.34	10.7
Pb	12.8	7.99	8.02	11.7	20.8	14.3
SiO ₂	4.87	2.46	2.83	4.09	5.14	6.26
S°	5.0	10.4	11.2	7.7	3.3	1.6
S(SO ₄)	7.36	3.78	2.57	4.01	7.65	10.2
S	19.5	31.9	34.4	28.3	15.1	11.1
Zn	16.2	31.1	33.8	26.3	12.2	6.20
Distribution, %						
Cu	100	24.0	9.4	14.5	9.6	42.5
Fe	100	18.6	6.8	11.0	8.5	55.0
Pb	100	12.8	4.6	10.8	14.7	57.2
Zn	100	39.3	15.2	19.1	6.8	19.6

Table 9.18 Batch Zinc Flotation: Supplementary Test 10 (Cleaner)

Feed Material	: Rougher concentrate (0 to 2 min) produced as in Test 9, diluted with filtrate
Solids Concentration	: 70 g/L slurry (2% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (20 g/L)
Temperature	: 60°C
Reagents	: Z200, 400 g/t, used for rougher only
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (140 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	1	2	5	-
Weight, g	143.5	54.7	27.0	13.7	48.1
Analysis, %					
Cd	0.11	0.12	0.14	0.14	0.07
Ca	2.09	<0.01	<0.01	<0.01	6.23
Cu	0.84	0.92	0.98	0.97	0.63
Fe	9.23	9.03	9.15	9.50	9.42
Pb	8.81	1.20	1.64	9.13	21.4
SiO ₂	2.62	0.57	0.79	3.21	5.81
S°	9.0	14.0	12.0	10.7	1.2
S(SO ₄)	3.79	0.52	0.62	2.10	9.76
S	31.2	42.6	41.3	34.9	11.4
Zn	30.1	44.0	43.5	33.0	6.02
Distribution, %					
Cu	100	41.8	22.0	11.0	25.2
Fe	100	37.3	18.7	9.8	34.2
Pb	100	5.2	3.5	9.9	81.4
Zn	100	55.7	27.2	10.5	6.7

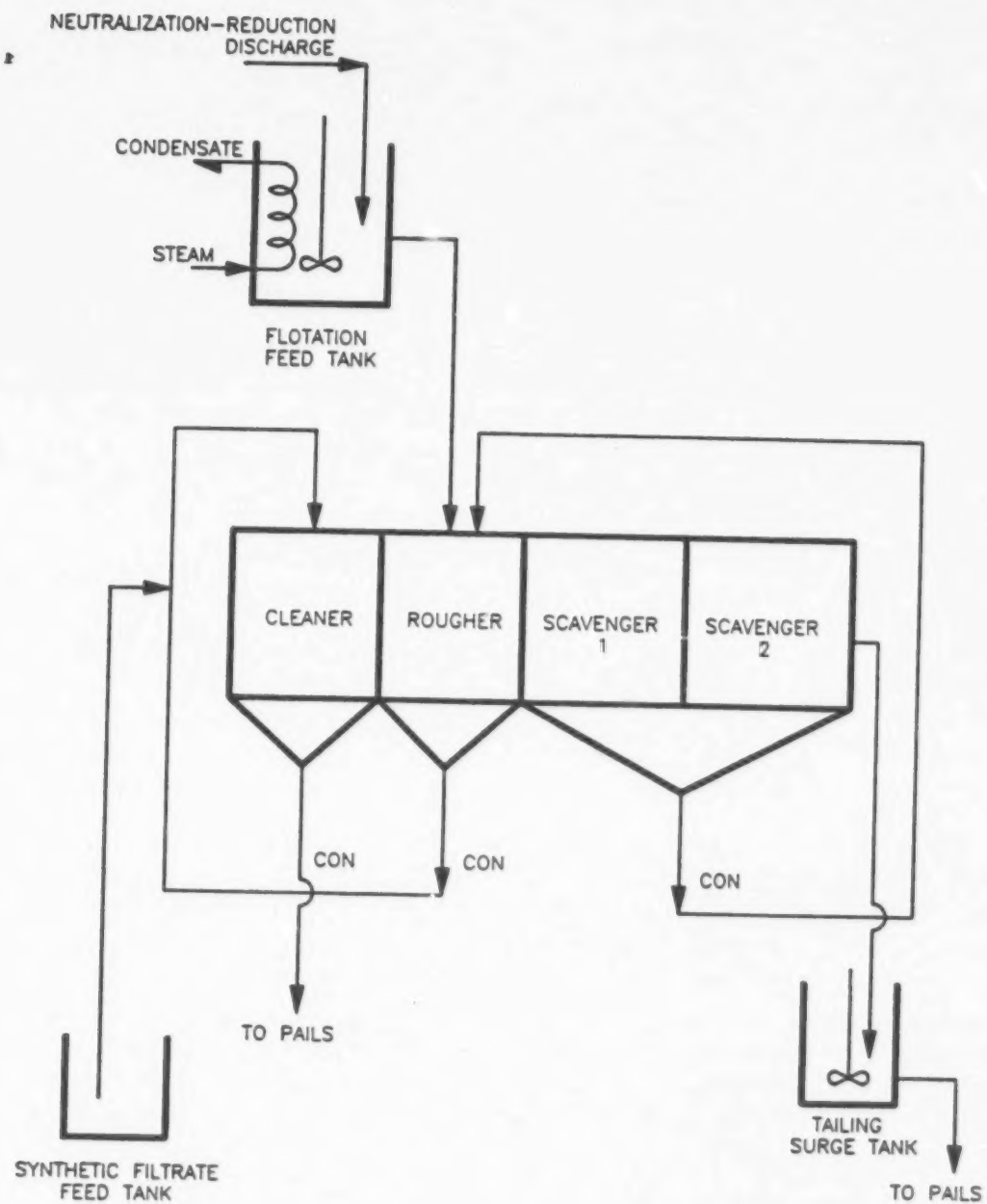


Figure 9.1 Continuous Zinc Flotation Equipment Flowsheet

10.0 REDUCING LEACH

Zinc flotation tailing was treated with sulphur dioxide and elemental sulphur in the reducing leach step. The process objectives were:

- to extract the remaining zinc, iron and copper from the solids;
- to reprecipitate copper as copper sulphide for recovery by flotation;
- to convert lead in the solids to lead sulphate so as to facilitate recovery of a clean lead concentrate; and
- to reduce iron in solution to the ferrous state for feed to the iron removal step.

Two batch reducing leach tests were performed prior to the continuous test to confirm the viability of the proposed flowsheet. A continuous reducing leach circuit was operated for 44 h with 100% on stream time. A total of 304 L of zinc flotation tailing slurry, containing 70 kg of solids, was processed through the circuit.

10.1 FEED MATERIALS

Zinc flotation tailing resulting from batch tests was the feed for the batch reducing leach tests. Continuous zinc flotation tailing was the feed for the continuous reducing leach test. The continuous flotation tailing was blended before distributing to pails in order to provide a uniform feed for the reducing leach. Feed samples were collected as part of the regular sampling profile during the reducing leach. The average analyses are summarized below. The slurry contained about 16% by weight solids.

Component	Analysis							
	As	Cd	Cu	Fe ²⁺	Fe	Pb	H ₂ SO ₄	Zn
Solution, g/L	2.20	0.15	3.62	17.6	21.1	-	22.6	81.6
Solids, %	0.53	0.07	0.60	-	10.7	17.4	-	7.03

Sulphur dioxide gas (Matheson) and flowers of sulphur (Fisher Scientific) were reagent grade. Sulphur was added batchwise to each pail of feed along with 0.1 mL of 300 g/L Lignosol solution as a wetting agent (750 g Lignosol per tonne elemental sulphur, or about 5 g/t oxide residue).

10.2 BATCH TESTS

Two batch reducing leach tests were conducted with zinc flotation tailing slurry generated from batch treatment of HBM&S oxide residue. Elemental sulphur was added to

help precipitate copper from solution. The leach conditions were 100°C and 100 kPa sulphur dioxide overpressure.

Copper was rapidly precipitated in the first test (Table 10.1) but the iron and zinc extraction rates were slow, possibly due to the high pulp density or ineffective agitation due to use of a radial turbine for the lower impeller.

Dual axial impellers were employed in the second test, and the solids concentration was decreased to 127 g/L slurry (Table 10.2). The results for this test are summarized below.

Retention Time, h	Solution Analysis, g/L			Solids Analysis, %				Extraction, % *		
	Cu	Fe	H ₂ SO ₄	Cu	Fe	Pb	Zn	Cu	Fe	Zn
Head	2.98	9.9	11.1	0.4	13.1	17.5	6.1	76	18	54
0.5	0.89	16.9	15.2	2.6	10.1	22.3	6.2	0	50	63
1.0	0.21	22.4	9.8	3.2	7.3	22.2	4.7	0	64	72
2.0	0.01	23.7	7.8	3.2	3.7	24.8	2.9	0	84	85
3.0	<0.01	25.5	6.7	3.6	2.1	23.2	2.0	0	90	89
4.0	<0.01	25.0	6.2	3.9	1.5	23.7	1.7	0	93	91

* with respect to original oxide residue head

Copper precipitation was essentially complete within 2 h, with only 0.01 g/L Cu remaining in solution. Iron and zinc extractions were levelling off at 4 h, but it would appear that some benefit might be gained from an extended retention time or a higher temperature. Higher iron extractions (95 to 98%) were obtained in the Phase Ia and Phase Ib process development studies, when two stages of reducing leaching were employed. The present overall zinc extraction was very similar to that obtained in the earlier testwork.

Ferrous iron concentrations in the test solutions, determined by titration, were invariably higher than the total iron analyses determined by ICP (see Tables 10.1 and 10.2). It is likely that sulphur dioxide dissolved in the solutions was interfering with the ferrous iron analyses. The ICP analyses are considered to be more reliable and it is likely that virtually all of the iron in the solutions was in the ferrous state.

10.3 CONTINUOUS REDUCING LEACH TEST

10.3.1 Circuit Operation

A detailed description of the equipment and procedures for the reducing leach circuit is provided in Section 6. The equipment flowsheet is provided in Figure 10.1. The

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leaching vessel was a titanium autoclave with a working volume of 30 L. The autoclave was divided into six compartments (C1 to C6). Elemental sulphur was blended into each pail of zinc flotation tailing prior to adding to the feed tank. The feed slurry was pumped continuously to C1. Sulphur dioxide was introduced to the vapour space in compartments C1 to C5. The gas was added on demand, to maintain the desired total pressure. Discharge from the autoclave passed through a chamber for recovery and recycle of sulphur dioxide. Slurry was pumped from the bottom of the chamber into pails. The discharge sample (DX) was collected at the point of entry to the pails.

At startup, the autoclave was filled with synthetic solution. Once at temperature, air was vented from the autoclave, sulphur dioxide overpressure was applied, and slurry was fed to C1. Analysis of the fill solution is provided below.

	Analysis, g/L				
	Cu	Fe ²⁺	Fe	H ₂ SO ₄	Zn
Autoclave Fill Solution	3.64	19.9	19.9	5.0	95.1

10.3.2 Sampling Procedures

Profile samples were taken every six hours from the feed tank, the six autoclave compartments and the discharge line (no C6 samples were collected for the first two profiles). The discharge slurry was sampled every hour for analysis of solutions which were titrated for Fe²⁺ and H₂SO₄ for control purposes. Copper and total iron analyses were determined later by ICP. The profile solutions were titrated for H₂SO₄ and later analyzed for As, Cd, Ca, Cu, Fe, Si and Zn by ICP. Profile solids samples were analyzed for As, Cd, Ca, Cu, Fe, Pb, SiO₂ and Zn.

10.3.3 Extraction Calculations

Metals extractions were calculated using a lead tie, as shown below for zinc. Overall extractions for the process were calculated, using the oxide residue analyses for the feed.

$$\% \text{ Zn Extraction} = \left[1 - \frac{\% \text{ Zn Residue}}{\% \text{ Zn Feed}} \times \frac{\% \text{ Pb Feed}}{\% \text{ Pb Residue}} \right] \times 100$$

As described in Sections 7.3.3 and 7.3.7, inclusion of the "water soluble" zinc portion of the oxide residue resulted in higher overall zinc extractions.

10.3.4 History of Events

Run Time h	Events and Changes
-1.0	Charged autoclave with 32 L of synthetic solution. Started heating autoclave under agitation.
-0.5	Vented air from autoclave; applied 100 kPa SO ₂ overpressure. Discharged excess solution from the last compartment (2 L).
0.0	Started feed slurry to autoclave: 115 mL/min. Elemental sulphur added to feed slurry at a rate of 4 g/L slurry.
7.5	Replaced leaking tubing in SO ₂ recycle pump head.
11.0	Decreased elemental sulphur feed rate to 2.67 g/L slurry
23.0	Decreased elemental sulphur feed rate to 1.33 g/L slurry. Autoclave discharge system changed. Discharge cooling coil removed; slurry pumped directly to the slurry-vapour separation chamber to allow for better flashing of sulphur dioxide in the discharge slurry. Slurry temperature now 74°C after discharge from the slurry-vapour separation chamber, compared with 33°C during previous operation.
25.5	Discharge has become thicker in appearance, more thixotropic. Pulp density has not changed.
26.0	Crack discovered in top flange of the slurry-vapour separation chamber. Autoclave vented to displace air that may have entered the system through this crack.
26.5	Changed the gasket at the top of the slurry-vapour separation chamber to give a better seal at the crack in the top flange. Autoclave discharge interrupted for 15 minutes while changing the gasket. No feed interruption.
29.0	Increased elemental sulphur feed rate to 2.67 g/L slurry.
44.0	Last sample profile taken. Feed slurry, sulphur dioxide, and gas burners shut off. Autoclave purged with nitrogen and vent gas passed through hydrogen peroxide scrubber.
45.0	Contents discharged from autoclave and interior examined. No solids build up or scale on autoclave internals; and no signs of corrosion.

10.3.5 Operating Parameters

The reducing leach operating parameters are given in Tables 10.3 and 10.4. A summary table is provided below

Period	Run Time h	Temp. °C	Pressure kPa (abs)	Ret. Time h	Sulphur g/L	Discharge Method
1	1 to 11	100	200	4.3	4.0	Precooled
2	11 to 23	100	200	4.3	2.67	Precooled
3	23 to 29	100	200	4.3	1.33	Flashed
4	29 to 44	100	200	4.4	2.67	Flashed

The temperature in the autoclave compartments ranged from 98 to 103°C, and was maintained at 100°C on average throughout the run. The total pressure was 200 kPa, with a sulphur dioxide overpressure of about 100 kPa. The feed rate was maintained between 114 and 116 mL/min of slurry, representing a retention time of 4.3 to 4.4 hours in the autoclave.

The only changes in the operations consisted of variation in the quantity of elemental sulphur added, and the method of slurry discharge. The sulphur addition rate varied between 1.33 and 4.0 g/L slurry, and was the basis for the division into 4 operating periods. Discharge slurry was passed through a cooling coil before entry into the slurry-vapour separation chamber in Periods 1 and 2. The slurry was discharged directly into this chamber from the last autoclave compartment in Periods 3 and 4.

10.3.6 Reagent Consumption

Consumption of elemental sulphur and sulphur dioxide in the reducing leach circuit is summarized below. The consumptions are indicated in terms of slurry feed rate (g/L) and oxide residue feed rate (kg/t). The latter is calculated from the solids feed rate to the reducing leach (0.230 kg solids per L slurry) and a lead tie, assuming that 98% of the lead in the oxide residue reports to the feed to the reducing leach (the remaining 2% reports to the zinc flotation concentrate).

Period	Run Time h	Sulphur		Sulphur Dioxide		Discharge Method
		g/L	kg/t	g/L	kg/t	
1	1 to 11	4.0	13.4	349	169	Precooled
2	11 to 23	2.67	9.0	326	158	Precooled
3	23 to 29	1.33	4.5	238	115	Flashed
4	29 to 44	2.67	9.0	239	117	Flashed

Recycle of the discharge vapour phase, containing excess sulphur dioxide, to the autoclave allowed for measurement of the quantity of SO_2 consumed by reaction in the reducing leach step. Changing to the flash discharge system in Period 3 had a large impact on the SO_2 consumption, which decreased to 115 kg/t oxide residue from 158 kg/t in Period 2. A significant quantity of sulphur dioxide is dissolved in the reducing leach solution if it is cooled under pressure, and less SO_2 is then available for recycle.

Dissolution of SO_2 to form sulphurous acid also affects the acid concentration of the solution, as indicated by the " H_2SO_4 " titration analyses (see Figure 10.2). The acid concentration in the discharge solution was analyzed as about 12 g/L H_2SO_4 on average in Period 2, compared with about 6 g/L H_2SO_4 in Periods 3 and 4. Flashing of the discharge results in less sulphur dioxide consumption in the reducing leach, and decreased lime or limestone consumption in the subsequent iron removal step.

The appearance of the reducing leach discharge also changed noticeably upon switching to the flash discharge system. The slurry became thicker in appearance (more thixotropic), although the pulp density was not affected.

The acid concentration in the discharge solution was abnormally high in the first few hours of operation owing to precipitation of copper in the fill solution, as described in Section 10.3.9.

Sulphur dioxide is consumed in the precipitation of copper, the dissolution of iron-bearing minerals remaining in the residue after the hot acid leach, and in the reduction of ferric iron remaining in solution after the neutralization-reduction step (see Section 4). The quantity of sulphur dioxide required for these latter two purposes can be reduced by more efficient operation of the previous process steps. A higher acid addition rate in the hot acid leach would lead to increased reaction in this first step, and a lower SO_2 consumption in the reducing leach. It should be noted that more than two thirds of the material discharged in the hot acid leach test was produced with a relatively low acid addition rate (see Section 7).

Similarly, the concentration of ferric iron in the reducing leach feed solution averaged 3.5 g/L. It may be possible to decrease this concentration to 1 or 2 g/L Fe^{3+} in neutralization-reduction without affecting the copper deportment. (Complete reduction of iron would likely coincide with some precipitation of copper from solution by metathesis with zinc sulphide.)

Decreasing the concentration of ferric iron in the feed solution by 1 g/L corresponds to a decrease in sulphur dioxide consumption of about 2 kg/t oxide residue. Consumption of sulphur dioxide in the precipitation of copper is approximately 10 kg/t for each percent of copper by weight in the oxide residue, or about 16 kg/t for the material treated (see below). Increasing the degree of reaction in the hot acid leach and ensuring efficient recycle of

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sulphur dioxide in the reducing leach would appear to be the most significant factors for minimizing sulphur dioxide consumption. Under optimum conditions, consumption of 100 kg/t or less would be expected.

Sulphur and sulphur dioxide are both consumed in the reducing leach in precipitating copper from solution, as shown in the following reaction.



The stoichiometric requirements of elemental sulphur and sulphur dioxide are both one mole per mole of copper precipitated. For 100% recovery of copper in the oxide residue feed to a copper sulphide precipitate, this would correspond to 8 kg elemental sulphur and 16 kg SO₂ per tonne of oxide residue (1.6% Cu in the oxide residue). It should also be noted that a portion of the copper in the zinc concentrate added during neutralization-reduction is leached during that step, and consumes additional sulphur and sulphur dioxide in the reducing leach. Copper from this source represented only about 5% of the copper in the feed to the reducing leach in the continuous testwork (see Section 4), but this fraction could increase if a lower grade zinc concentrate were to be used in neutralization-reduction.

The feed rate of copper to the reducing leach is compared with the elemental sulphur addition rate in the table below. The feed solution contained 3.62 g/L Cu on average; the feed solids 0.60% Cu.

Period	1	2	3	4
Feed Solution, L/h	6.54	6.54	6.53	6.47
Cu in Solution, g/h	23.7	23.7	23.6	23.4
Feed Solids, kg/h	1.59	1.59	1.59	1.58
Cu in Solids, g/h	9.5	9.5	9.5	9.4
Total Cu, g/h	33.2	33.2	33.1	32.8
Sulphur Addition, g/h	27.8	18.5	9.2	18.3
S: Cu Molar Ratio	1.7	1.1	0.6	1.1

The rate of elemental sulphur addition was 4 g/L of slurry or 13.4 kg/t of oxide residue in the first period of operation. This corresponded to 1.7 moles of elemental sulphur per mole of copper in the feed to the reducing leach. Copper was readily precipitated from solution to a concentration of only 0.01 g/L Cu at this rate, and the sulphur addition rate was decreased by 33% in Period 2. Copper in the discharge solution did not increase in Period 2, and the sulphur addition rate was decreased by 50% in Period 3. Although this represented what was thought to be a substoichiometric addition of sulphur, (see table above), copper continued to be precipitated essentially quantitatively from the solution. Elemental sulphur analyses of the continuous zinc flotation products obtained after completion of the reducing leach test revealed that zinc flotation tailing

solids contained 3.4% S° on average. This represented an addition of about 50 g/h S° to the reducing leach, which is about three times the stoichiometric requirement for precipitating copper, suggesting that copper would likely have been precipitated just as effectively during the reducing leach if no additional sulphur was added.

As described in Section 9, the scavenger flotation was not very efficient in the continuous zinc flotation test, and the result was that high levels of zinc and elemental sulphur reported to the continuous zinc flotation tailing solids. These solids would be expected to contain only 0.5% to 1.0% S° under optimum flotation conditions, and the addition of elemental sulphur to the reducing leach would be required. The elemental sulphur addition would likely be in the range of 5 to 10 kg per tonne of oxide residue treated (for a head analysis of 1.6% Cu in the oxide residue).

10.3.7 Copper Deportment

Solution analyses for the continuous reducing leach test are given in Tables 10.5 to 10.8; and residue analyses in Tables 10.9 and 10.10. Copper concentration in the autoclave discharge is plotted as a function of run time in Figure 10.3. Copper precipitation was very successful, and with the exception of the first few hours of operation, the solution concentration averaged 0.01 g/L Cu or less in the discharge. The retention time of 4.3 h was longer than required for precipitation of virtually all of the copper. The average copper analyses for the solution profile samples for the run as a whole are given in Figure 10.4 and in the table below. About 90% of the copper was precipitated in the first compartment; and most of the remainder in the second. A retention time of 1 to 2 hours would appear to be sufficient for copper precipitation.

Sample	Feed	C1	C2	C3	C4	C5	C6	DX
Copper in Sol'n, g/L	3.62	0.43	0.04	0.02	0.01	0.01	0.01	0.01

The average concentration of copper in the solids is given for each sample in the profile in the table below. Although virtually all of the copper was precipitated from solution in the first two compartments, the copper concentration in the solids continued to increase in the remaining compartments as other minerals dissolved and the weight of solids decreased. The average discharge (DX) analysis does not include the value of 7.82% Cu obtained in the first profile. This concentration was abnormally high owing to the precipitation of copper from the autoclave fill solution.

Sample	Feed	C1	C2	C3	C4	C5	C6	DX
Copper in Solids, %	0.60	2.18	2.62	2.85	2.95	3.04	3.10	3.13

10.3.8 Iron and Zinc Extractions

Residue analyses for the continuous reducing leach test are given in Tables 10.9 and 10.10. Iron and zinc extractions in the autoclave discharge sample are plotted in Figure 10.5, and both analyses and extractions are summarized in the table below. The analyses at 5 h run time are not included in the averages as the system had not yet reached equilibrium.

Run Time h	DX Analysis			DX Extraction		Corrected* Zn Extraction %
	Fe %	Pb %	Zn %	Fe %	Zn %	
5	1.89	21.3	4.36	91.6	75.6	77.2
11	1.70	23.0	2.96	93.0	84.7	85.7
17	1.77	24.4	2.96	93.1	85.5	86.5
23	2.04	22.5	3.21	91.4	83.0	84.1
29	2.61	25.1	3.46	90.2	83.6	84.7
35	2.73	28.6	3.95	91.0	83.5	84.6
41	2.05	24.8	3.30	92.2	84.1	85.2
44	2.18	26.7	3.45	92.3	84.6	85.6
Average	2.15	25.0	3.33	91.9	84.2	85.2

* including "water soluble" zinc component

Overall iron and zinc extractions with respect to the oxide residue feed solids averaged 92% and 84%, respectively. If the "water soluble" zinc component of the oxide residue feed is considered, that is the zinc contained in the solution entrained in the oxide residue filter cake, the overall zinc extraction is increased by about 1%.

Iron and zinc extractions in Batch Test 2 were 93% and 91%, respectively, after 4 h retention time. The iron extraction in the continuous tests is comparable, but the zinc extraction is significantly lower than in the batch test. The batch discharge solids contained only 1.7% Zn compared with 3.3% Zn in the continuous test. The higher zinc concentration in the continuous test resulted from incomplete separation of zinc sulphide in the continuous zinc flotation step. Zinc sulphide in the zinc flotation tailing survives the reducing leach and leads to a high zinc concentration and an apparent low overall zinc extraction in the reducing leach discharge solids.

Residual zinc sulphide in the reducing leach discharge is collected along with copper sulphide in the copper flotation step. The continuous copper flotation tailing

contained only 1.3 % Zn on average, indicating an overall zinc extraction of 94 % (see Section 11).

Average iron and zinc extraction profiles for the continuous reducing leach test (excluding values for the first profile, when equilibrium had not yet been attained) are given in Figure 10.6 and the data are summarized in the table below. Both iron and zinc extractions were complete by the fifth autoclave compartment, indicating that the retention time of about 4 hours was sufficient.

Sample	Analysis, %			Extraction, %		Corrected* Zn Extraction, %
	Fe	Pb	Zn	Fe	Zn	
Feed	10.7	17.4	7.03	41.9	51.9	55.0
C1	7.40	19.6	5.52	64.2	66.3	68.5
C2	4.86	21.6	4.33	78.7	76.1	77.7
C3	3.57	23.2	3.83	85.4	80.3	81.6
C4	2.64	23.7	3.43	89.4	82.8	83.9
C5	2.38	24.1	3.36	90.6	83.3	84.4
C6	2.58	24.8	3.55	90.2	83.0	84.1
DX	2.15	25.0	3.33	91.9	84.2	85.2

* including "water soluble" zinc component

10.3.9 Solution Quality

Acid, copper, and iron and zinc concentrations in the discharge solution are plotted against run time in Figures 10.2, 10.3 and 10.7, respectively. Once equilibrium was reached, the metals concentrations in solution were reasonably stable, averaging 0.01 g/L Cu, 43 g/L Fe and 91 g/L Zn. As described in Section 10.2, ferrous iron analyses were invariably higher than the total iron analyses. It is likely that sulphur dioxide dissolved in the solutions was interfering with the ferrous iron analyses. The ICP analyses are considered to be more reliable and it is likely that virtually all of the iron in the discharge solution was in the ferrous state.

As described in Section 10.3.6, the acid concentration in the discharge solution decreased after 23 h run time when the flash discharge system was installed. This difference is also evident in the solution profiles, as shown in Figures 10.8 and 10.9, and in the table overleaf.

Since the autoclave compartment samples were obtained by flashing slurry into a collection vessel at atmospheric pressure, the quantity of sulphur dioxide dissolved in these samples was minimized. The autoclave discharge was cooled prior to separation of the vapour for the first 23 hours of operation, and the discharge samples obtained during

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this time contained about 5 or 6 g/L H_2SO_4 (or equivalent as H_2SO_3) more than the last few compartments of the autoclave.

Sample	0 to 23 h		23 to 44 h	
	Analysis, g/L		Analysis, g/L	
	Fe	H_2SO_4	Fe	H_2SO_4
Feed	21.1	22.6	21.1	22.6
C1	33.6	21.6	30.8	21.4
C2	40.0	14.1	37.6	12.2
C3	42.6	9.8	41.1	8.1
C4	44.3	8.6	41.9	7.1
C5	44.9	8.8	42.6	6.4
C6	46.2	6.3	42.8	5.1
DX	41.6	13.9	42.6	5.6

The acid concentration in the discharge solution was abnormally high during the first few hours of operation (see Figure 10.2). This excess acid corresponded with the acid generated in precipitating copper in the autoclave fill solution. Precipitation of all the copper in the fill solution would be expected to generate 11 g/L H_2SO_4 according to the reaction given in Section 10.3.6.

With the flash discharge in place, the reducing leach solution contained about 5 g/L " H_2SO_4 " and 43 g/L iron, in the ferrous state. This solution is suitable as feed to the iron removal step, where iron is precipitated as ferric hydroxide (see Section 13).

10.4 Conclusions and Recommendations

A retention time of 4 h, at 100°C and under 100 kPa sulphur dioxide overpressure is sufficient to meet all of the process objectives in the reducing leach.

Net iron extraction from the stockpiled oxide residue is about 92%. Net zinc extraction, including zinc recovered with the zinc flotation concentrate, was about 85% in the continuous testwork. Zinc extraction would be expected to increase to about 94% with improved scavenging of zinc sulphide in the zinc flotation step.

Precipitation of copper is essentially quantitative, with only 0.01 g/L Cu in the discharge solution. Iron in the discharge solution is virtually all in the ferrous state.

The reagent requirements are about 10 kg of elemental sulphur and 100 kg of sulphur dioxide per tonne of stockpiled oxide residue. Flashing of the reducing leach discharge slurry is recommended to minimize reagent requirements in the reducing leach and iron removal circuits.

Table 10.1 Reducing Leach: Batch Test 1

Charge	: Scavenger tailing from batch zinc flotation Test 2 repulped to 2.0 L with 10 g/L H ₂ SO ₄ solution
Solids Concentration	: 234 g/L slurry
Temperature	: 100°C
Additives	: Sulphur, 5 g (10 kg/t)
Reductant	: SO ₂ , added to maintain constant pressure
Total Pressure	: 200 kPa
SO ₂ Overpressure	: 100 kPa
Equipment	: 3.8 L titanium-lined autoclave, equipped with baffles and gas inlet tube to the vapour space.
Agitation	: Axial over radial impellers, tip speed 4.5 m/s

	Retention Time, h				
	Head	0.5	1.0	2.0	4.0
Solutions, g/L					
As	0.85	1.52	1.76	1.97	2.15
Cd	0.24	0.28	0.31	0.33	0.31
Cu	2.14	0.09	0.01	0.01	<0.005
Fe ²⁺	3.44	15.5	24.8	26.2	31.5
Fe	5.60	12.1	16.8	20.6	21.3
H ₂ SO ₄	8.60	12.9	9.0	5.6	4.9
Zn	31.8	37.0	41.6	44.6	41.4
Residues, %					
As	3.08	3.29	3.53	3.77	3.18
Cd	0.06	0.07	0.07	0.07	0.06
Ca	5.90	6.19	6.18	6.78	6.52
Cu	0.80	1.94	2.17	2.21	2.24
Fe	11.3	10.2	9.17	7.86	6.72
Pb	18.9	22.9	24.7	25.3	25.3
SiO ₂	2.98	4.56	5.21	6.03	4.91
Zn	4.65	4.09	3.43	2.82	2.57
Extraction*, %					
Fe	13	35	46	55	61
Zn	56	68	75	80	82

* With respect to oxide residue feed, based on lead tie.

Table 10.2 Reducing Leach: Batch Test 2

Charge	:	Wet, unwashed tailing from batch zinc flotation Test 3 repulped to 2.0 L with 10 g/L H ₂ SO ₄ solution
Solids Concentration	:	127 g/L slurry
Temperature	:	100°C
Additives	:	Sulphur, 5 g (20 kg/t)
Reductant	:	SO ₂ , added to maintain constant pressure
Total Pressure	:	200 kPa
SO ₂ Overpressure	:	100 kPa
Equipment	:	3.8 L titanium-lined autoclave, equipped with baffles and gas inlet tube to the vapour space.
Agitation	:	Dual axial impellers, tip speed 4.5 m/s

	Retention Time, h					
	Head	0.5	1.0	2.0	3.0	4.0
Solutions, g/L						
As	1.46	2.38	2.75	2.46	2.44	2.38
Cd	0.16	0.18	0.21	0.19	0.20	0.19
Ca	0.57	0.67	0.74	0.57	0.61	0.59
Cu	2.98	0.89	0.21	0.01	<0.005	<0.005
Fe ²⁺	3.7	20.6	24.5	28.8	31.2	30.6
Fe	9.85	16.9	22.4	23.7	25.5	25.0
H ₂ SO ₄	11.1	15.2	9.8	7.8	6.7	6.2
Zn	47.7	50.8	58.7	54.0	54.6	52.4
Residues, %						
As	0.88	0.32	0.23	0.21	0.16	0.16
Cd	0.07	0.08	0.07	0.07	0.06	0.06
Ca	7.94	7.63	8.09	9.76	10.4	11.2
Cu	0.44	2.55	3.24	3.20	3.61	3.92
Fe	13.1	10.1	7.29	3.73	2.11	1.47
Pb	17.5	22.3	22.2	24.8	23.2	23.7
SiO ₂	4.64	8.12	8.14	8.51	8.22	8.46
Zn	6.07	6.23	4.68	2.89	2.00	1.69
Extraction*, %						
Cu	76	0	0	0	0	0
Fe	18	50	64	84	90	93
Zn	54	63	72	85	89	91

* With respect to oxide residue feed, based on lead tie.

Table 10.3 Continuous Reducing Leach Operating Parameters (0 to 29 h)

Run Time h	Slurry Feed mL/min	Sulphur Addition g/L slurry	SO ₂ Rate g/h	Total Pressure kPa	Temperature, °C						Discharge	
					C1	C2	C3	C4	C5	C6	Rate mL/min	P.D. g/L
1	115	4.0	388	200	100	99	100	100	98	99	115	n.a.
2	115	4.0	349	200	100	99	100	100	98	99	120	n.a.
3	116	4.0	349	200	100	99	102	102	99	100	120	n.a.
4	116	4.0	344	200	100	99	102	103	99	99	120	n.a.
5	116	4.0	366	200	99	99	102	102	100	98	124	n.a.
6	117	4.0	339	200	100	99	101	100	100	99	122	1474
7	116	4.0	334	200	100	99	102	101	100	100	128	1485
8	115	4.0	360	200	100	101	101	100	100	100	118	1480
9	116	4.0	354	200	100	99	101	100	100	100	118	1483
10	115	4.0	317	200	100	100	102	101	101	100	112	1473
11	116	4.0	335	200	100	101	103	102	102	100	116	1479
12	115	2.67	338	200	100	100	103	102	102	100	113	1468
13	116	2.67	347	200	100	100	103	102	102	100	118	1478
14	115	2.67	354	200	100	100	101	101	101	100	120	1476
15	116	2.67	364	200	100	100	100	100	100	100	119	1476
16	115	2.67	340	200	100	100	101	100	100	100	115	1481
17	115	2.67	344	200	101	100	101	100	101	100	115	1477
18	117	2.67	321	200	100	100	102	100	100	100	117	1471
19	115	2.67	309	200	100	100	100	100	101	100	117	1475
20	115	2.67	321	200	100	100	100	99	100	100	114	1482
21	116	2.67	300	200	100	100	100	99	100	100	115	1486
22	116	2.67	291	200	100	99	100	99	100	99	117	1473
23	116	2.67	284	200	100	100	101	99	100	100	116	1463
24	115	1.33	204	200	100	99	101	99	100	99	115	1470
25	116	1.33	200	200	100	100	101	99	100	100	114	1474
26	116	1.33	316	200	100	100	101	100	100	100	116	1486
27	115	1.33	238	200	100	99	101	100	100	100	117	1473
28	115	1.33	235	200	100	100	101	100	100	100	115	1475
29	116	1.33	236	200	100	100	101	100	100	100	117	1459

n.a. = not analyzed

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Table 10.4 Continuous Reducing Leach Operating Parameters (30 to 44 h)

Run Time h	Slurry Feed mL/min	Sulphur Addition g/L slurry	SO ₂ Rate g/h	Total Pressure kPa	Temperature, °C						Discharge	
					C1	C2	C3	C4	C5	C6	Rate mL/min	P.D. g/L
30	115	2.67	265	200	100	99	101	100	100	100	115	1479
31	116	2.67	260	200	100	99	102	100	100	99	112	1474
32	115	2.67	254	200	100	100	102	100	100	100	116	1481
33	115	2.67	255	200	100	100	102	100	100	100	112	1465
34	116	2.67	228	200	100	100	102	100	100	100	108	1464
35	114	2.67	230	200	100	100	102	100	100	100	111	1473
36	113	2.67	239	200	100	100	102	99	100	100	117	1471
37	121	2.67	228	200	100	100	102	99	100	100	121	1468
38	105	2.67	225	200	100	100	101	99	99	100	111	1470
39	112	2.67	231	200	101	100	101	99	100	100	117	1454
40	114	2.67	233	200	100	100	101	99	99	100	115	1456
41	117	2.67	236	200	99	100	100	98	99	99	121	1446
42	115	2.67	230	200	100	100	100	98	99	100	121	1464
43	115	2.67	231	200	100	100	100	98	99	100	117	1461
44	114	2.67	247	200	101	100	100	99	98	99	114	1456

Table 10.5 Reducing Leach Control Solution Analyses (0 to 29 h)

Run Time h	Sample	Analysis, g/L			
		Cu	Fe ²⁺	Fe	H ₂ SO ₄
1	DX	1.71	28.8	21.4	31.6
2	DX	1.26	27.9	24.1	34.0
3	DX	0.06	36.9	26.7	32.2
4	DX	0.07	43.9	31.7	26.4
5	DX	0.01	44.6	36.3	21.3
6	DX	0.01	49.7	38.3	16.2
7	DX	0.01	58.5	42.0	15.3
8	DX	0.01	57.1	41.8	13.0
9	DX	0.01	63.8	44.1	13.7
10	DX	0.01	58.2	43.5	12.2
11	DX	0.01	53.5	40.0	11.2
12	DX	0.01	56.2	40.5	12.3
13	DX	0.01	64.5	45.5	12.7
14	DX	0.01	61.0	44.3	13.0
15	DX	0.01	55.5	39.0	11.1
16	DX	0.01	43.7	43.3	11.7
17	DX	0.01	60.2	45.1	12.9
18	DX	0.02	58.0	44.5	10.6
19	DX	0.01	60.0	43.3	11.6
20	DX	0.01	58.7	42.9	11.4
21	DX	0.01	60.0	42.5	11.9
22	DX	0.02	59.8	46.4	11.2
23	DX	0.02	57.3	45.1	10.2
24	DX	0.01	49.3	45.3	5.5
25	DX	0.02	50.0	45.0	5.9
26	DX	0.03	49.0	43.3	6.0
27	DX	0.02	49.3	53.5	6.1
28	DX	0.02	50.3	43.2	6.1
29	DX	0.01	51.8	43.1	5.9

Table 10.6 Reducing Leach Control Solution Analyses (30 to 44 h)

Run Time h	Sample	Analysis, g/L			
		Cu	Fe ²⁺	Fe	H ₂ SO ₄
30	DX	0.02	48.3	41.6	5.3
31*	DX	<0.01	48.0	42.2	5.7
32	DX	<0.01	48.3	42.4	5.3
33	DX	<0.01	48.8	42.5	5.3
34	DX	0.02	44.2	38.4	4.6
35	DX	0.01	47.2	42.7	5.2
36	DX	<0.01	38.5	34.0	4.2
37	DX	<0.01	44.0	37.8	5.0
38	DX	0.03	43.2	37.4	4.9
39	DX	0.01	48.5	41.5	5.8
40	DX	<0.01	48.2	42.2	5.7
41	DX	0.01	49.0	40.8	6.0
42	DX	<0.01	45.5	40.4	5.6
43	DX	<0.01	51.2	44.8	5.9
44	DX	<0.01	47.7	43.6	5.3

Table 10.7 Reducing Leach Profile Solution Analyses (0 to 23 h)

Run Time h	Sample	Analysis, g/L							
		As	Cd	Ca	Cu	Fe	SiO ₂	H ₂ SO ₄	Zn
5	Feed	2.31	0.16	0.49	3.69	21.4	0.23	20.3	84.9
	C1	2.89	0.20	0.55	0.18	35.9	0.30	20.1	97.3
	C2	2.91	0.21	0.52	0.02	38.8	0.30	15.5	97.2
	C3	2.90	0.21	0.53	0.01	40.4	0.31	11.7	96.5
	C4	3.08	0.22	0.51	0.01	42.7	0.30	11.5	102.3
	C5	3.02	0.22	0.50	<0.01	40.8	0.30	13.7	101.2
	DX	2.84	0.20	0.40	0.01	36.3	0.29	21.3	97.4
11	Feed	2.29	0.16	0.42	3.62	21.4	0.15	25.0	82.3
	C1	2.75	0.19	0.47	0.19	34.2	0.21	20.1	90.8
	C2	2.98	0.23	0.50	0.05	40.3	0.28	13.8	96.1
	C3	3.05	0.22	0.47	0.03	43.8	0.27	8.9	97.3
	C4	3.11	0.22	0.48	0.01	44.7	0.26	7.3	96.2
	C5	3.20	0.22	0.46	0.01	45.4	0.26	7.2	95.9
	DX	2.83	0.20	0.30	0.01	40.0	0.22	11.2	84.5
17	Feed	2.35	0.16	0.42	3.76	22.0	0.11	24.9	85.2
	C1	2.68	0.18	0.47	0.45	31.6	0.19	21.7	88.1
	C2	2.96	0.21	0.50	0.02	40.8	0.24	12.9	96.8
	C3	3.02	0.01	0.49	0.01	43.8	0.24	8.9	97.8
	C4	3.10	0.22	0.50	0.01	45.4	0.24	7.5	98.1
	C5	3.32	0.23	0.50	0.01	47.8	0.25	7.1	101.9
	C6	3.27	0.23	0.49	0.01	46.5	0.27	6.5	97.7
	DX	3.17	0.22	0.32	0.01	45.1	0.24	12.9	94.8
23	Feed	2.44	0.18	0.47	3.91	22.9	0.19	20.8	88.1
	C1	2.80	0.19	0.52	0.59	32.8	0.27	24.4	92.7
	C2	2.97	0.21	0.52	0.03	40.2	0.28	14.3	96.3
	C3	2.96	0.21	0.53	0.02	42.6	0.33	9.9	95.7
	C4	3.02	0.22	0.52	0.02	44.5	0.30	8.3	97.2
	C5	3.12	0.22	0.53	0.02	45.7	0.32	7.3	97.9
	C6	3.19	0.22	0.54	0.02	46.0	0.30	6.1	97.3
	DX	3.12	0.22	0.38	0.02	45.1	0.29	10.2	95.4

Table 10.8 Reducing Leach Profile Solution Analyses (24 to 44 h)

Run Time h	Sample	Analysis, g/L							
		As	Cd	Ca	Cu	Fe	SiO ₂	H ₂ SO ₄	Zn
29	Feed	2.28	0.16	0.44	3.82	21.8	0.11	21.9	85.7
	C1	2.74	0.18	0.48	0.78	33.1	0.20	22.6	93.4
	C2	2.87	0.20	0.47	0.11	38.7	0.21	15.1	95.9
	C3	2.94	0.21	0.47	0.02	42.8	0.22	8.8	98.4
	C4	2.91	0.22	0.49	0.01	43.8	0.21	7.7	97.7
	C5	2.93	0.21	0.51	0.01	44.3	0.23	6.7	96.9
	C6	2.81	0.19	0.56	0.01	42.5	0.48	5.5	92.2
	DX	2.85	0.19	0.59	0.01	43.1	0.51	5.9	93.1
35	Feed	2.09	0.15	0.42	3.63	20.8	0.09	24.2	79.7
	C1	2.50	0.17	0.46	0.43	30.9	0.15	21.7	85.9
	C2	2.66	0.19	0.53	0.02	38.0	0.23	12.0	90.2
	C3	2.71	0.18	0.63	<0.01	40.9	0.40	7.9	91.5
	C4	2.71	0.18	0.58	0.01	41.1	0.49	7.2	89.7
	C5	2.73	0.18	0.61	0.01	41.8	0.42	6.2	89.7
	C6	2.81	0.19	0.56	0.01	42.9	0.36	4.9	91.6
	DX	2.83	0.19	0.62	0.01	42.7	0.31	5.2	90.2
41	Feed	1.75	0.12	0.38	3.11	17.9	0.08	20.8	68.4
	C1	2.21	0.15	0.42	0.38	27.7	0.13	19.5	76.9
	C2	2.35	0.16	0.67	0.02	34.5	0.40	10.1	80.4
	C3	2.54	0.18	0.65	0.01	39.0	0.28	7.8	86.6
	C4	2.53	0.18	0.64	0.01	39.2	0.27	6.6	85.1
	C5	2.59	0.18	0.63	0.01	40.0	0.23	6.5	84.9
	C6	2.56	0.18	0.65	<0.01	39.6	0.24	4.8	83.6
	DX	2.59	0.18	0.61	0.01	40.8	0.47	6.0	87.0
44	Feed	2.13	0.14	0.40	3.45	20.5	0.06	23.1	78.5
	C1	2.63	0.16	0.49	0.40	31.7	0.15	21.6	87.2
	C2	2.79	0.17	0.44	0.02	39.1	0.40	11.6	90.3
	C3	2.85	0.18	0.47	<0.01	41.7	0.27	7.9	91.3
	C4	2.92	0.18	0.45	<0.01	43.4	0.27	6.9	92.2
	C5	2.98	0.18	0.44	<0.01	44.4	0.28	6.4	91.8
	C6	3.11	0.19	0.45	<0.01	46.0	0.24	5.2	93.8
	DX	2.95	0.18	0.48	<0.01	43.6	0.30	5.3	88.5

Table 10.9 Reducing Leach Profile Solids Analyses (0 to 23 h)

Run Time h	Sample	Analysis, %							
		As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn
5	Feed	0.53	0.07	7.94	0.64	10.8	17.5	5.04	7.20
	C1	0.34	0.06	8.80	2.30	6.17	19.5	4.61	4.70
	C2	0.31	0.06	9.05	2.59	4.80	20.3	5.01	4.21
	C3	0.34	0.06	9.63	2.79	3.23	21.3	6.47	3.60
	C4	0.24	0.05	9.58	3.00	2.26	21.4	5.61	3.09
	C5	0.18	0.05	9.45	3.42	1.67	22.0	6.17	2.88
	DX	0.16	0.06	8.10	7.82	1.89	21.3	8.34	4.36
11	Feed	0.57	0.07	7.83	0.60	10.9	17.3	4.93	7.09
	C1	0.35	0.06	8.34	2.30	6.53	20.0	5.59	5.06
	C2	0.34	0.06	8.99	2.46	4.71	21.1	5.87	4.11
	C3	0.31	0.06	9.41	2.62	3.09	21.1	6.11	3.41
	C4	0.27	0.06	9.74	2.69	2.32	22.1	6.36	3.09
	C5	0.22	0.06	9.75	2.80	1.86	22.7	6.64	2.98
	DX	0.18	0.06	9.46	3.00	1.70	23.0	6.81	2.96
17	Feed	0.47	0.07	7.65	0.57	10.1	16.5	4.78	6.77
	C1	0.43	0.07	8.61	2.18	7.54	19.8	5.72	5.71
	C2	0.36	0.07	9.57	2.49	4.47	20.7	5.96	4.03
	C3	0.36	0.07	9.79	2.74	3.30	21.5	6.41	3.64
	C4	0.33	0.07	10.0	2.75	2.54	22.3	6.62	3.29
	C5	0.27	0.07	10.0	2.85	2.04	23.0	6.88	3.16
	C6	0.26	0.06	10.1	2.93	1.77	23.3	7.05	3.09
	DX	0.24	0.07	9.49	2.93	1.77	24.4	7.22	2.96
23	Feed	0.54	0.07	7.90	0.60	10.8	18.0	5.10	7.23
	C1	0.39	0.07	8.69	2.05	7.58	19.4	5.49	5.67
	C2	0.36	0.07	8.79	2.54	4.98	21.2	6.17	4.47
	C3	0.32	0.06	9.28	2.72	3.57	21.2	6.32	3.86
	C4	0.29	0.06	9.53	2.66	2.73	21.6	6.21	3.33
	C5	0.24	0.06	9.42	2.81	2.31	22.3	6.49	3.25
	C6	0.22	0.06	9.75	2.87	1.99	22.1	6.56	3.19
	DX	0.22	0.06	9.56	2.85	2.04	22.5	6.60	3.21

Table 10.10 Reducing Leach Profile Solids Analyses (24 to 44 h)

Run Time h	Sample	Analysis, %							
		As	Cd	Ca	Cu	Fe	Pb	SiO ₂	Zn
29	Feed	0.50	0.07	7.83	0.60	10.8	17.4	3.30	7.02
	C1	0.35	0.07	8.26	2.01	7.49	19.4	5.53	5.51
	C2	0.35	0.06	8.96	2.43	5.20	20.5	5.81	4.35
	C3	0.32	0.06	9.34	2.63	3.67	21.0	6.11	3.65
	C4	0.32	0.06	9.62	2.64	2.93	21.3	6.19	3.22
	C5	0.35	0.06	9.55	2.72	2.60	21.7	6.49	3.17
	C6	0.31	0.07	8.22	3.27	2.72	25.7	7.09	3.60
	DX	0.32	0.07	8.66	3.16	2.61	25.1	6.92	3.46
35	Feed	0.55	0.07	8.22	0.58	10.6	17.5	4.65	6.87
	C1	0.38	0.07	8.63	2.11	7.44	19.6	5.36	5.51
	C2	0.37	0.06	9.28	2.56	4.71	21.0	5.91	4.26
	C3	0.36	0.07	8.49	3.03	3.72	24.3	6.73	4.01
	C4	0.32	0.08	7.97	3.37	2.62	27.2	7.52	3.82
	C5	0.32	0.08	7.50	3.34	2.84	26.9	7.24	3.85
	C6	0.41	0.08	7.15	3.32	3.22	26.6	7.20	3.99
	DX	0.33	0.08	7.25	3.53	2.73	28.6	7.59	3.95
41	Feed	0.56	0.07	8.01	0.59	10.6	17.0	4.63	7.01
	C1	0.37	0.07	8.45	2.21	7.45	19.1	4.99	5.60
	C2	0.34	0.07	8.71	2.86	4.68	22.5	6.28	4.36
	C3	0.35	0.07	9.04	3.01	3.51	23.9	6.79	3.88
	C4	0.23	0.07	10.4	3.15	2.06	24.8	7.07	3.30
	C5	0.32	0.07	10.1	3.08	2.40	25.2	7.09	3.50
	C6	0.34	0.07	9.80	3.02	2.91	24.6	6.84	3.66
	DX	0.25	0.07	10.6	3.12	2.05	24.8	7.14	3.30
44	Feed	0.54	0.07	7.42	0.61	10.9	17.9	4.52	7.01
	C1	0.34	0.06	8.37	2.24	7.76	19.6	5.10	5.61
	C2	0.36	0.08	7.76	3.02	5.28	24.3	6.47	4.76
	C3	0.36	0.08	8.07	3.26	4.12	26.4	7.26	4.33
	C4	0.30	0.08	8.36	3.35	3.28	26.8	7.41	3.93
	C5	0.26	0.07	9.39	3.29	2.61	26.6	7.20	3.62
	C6	0.21	0.07	10.6	3.17	2.07	25.1	6.90	3.30
	DX	0.18	0.07	9.49	3.35	2.18	26.7	7.26	3.45

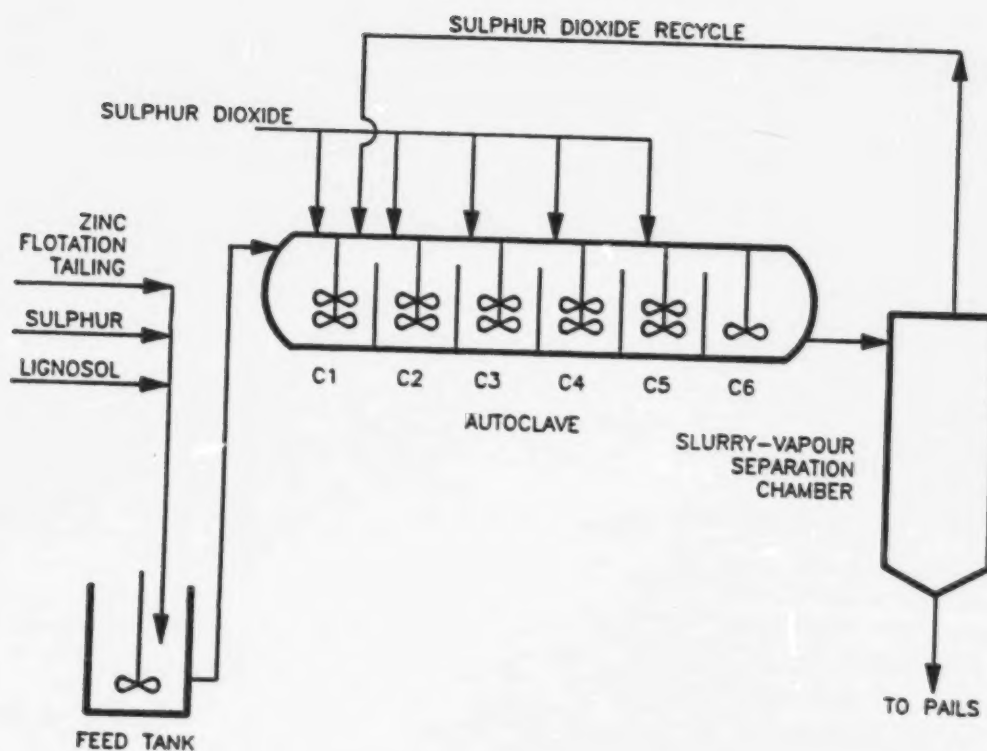


Figure 10.1 Continuous Reducing Leach Equipment Flowsheet

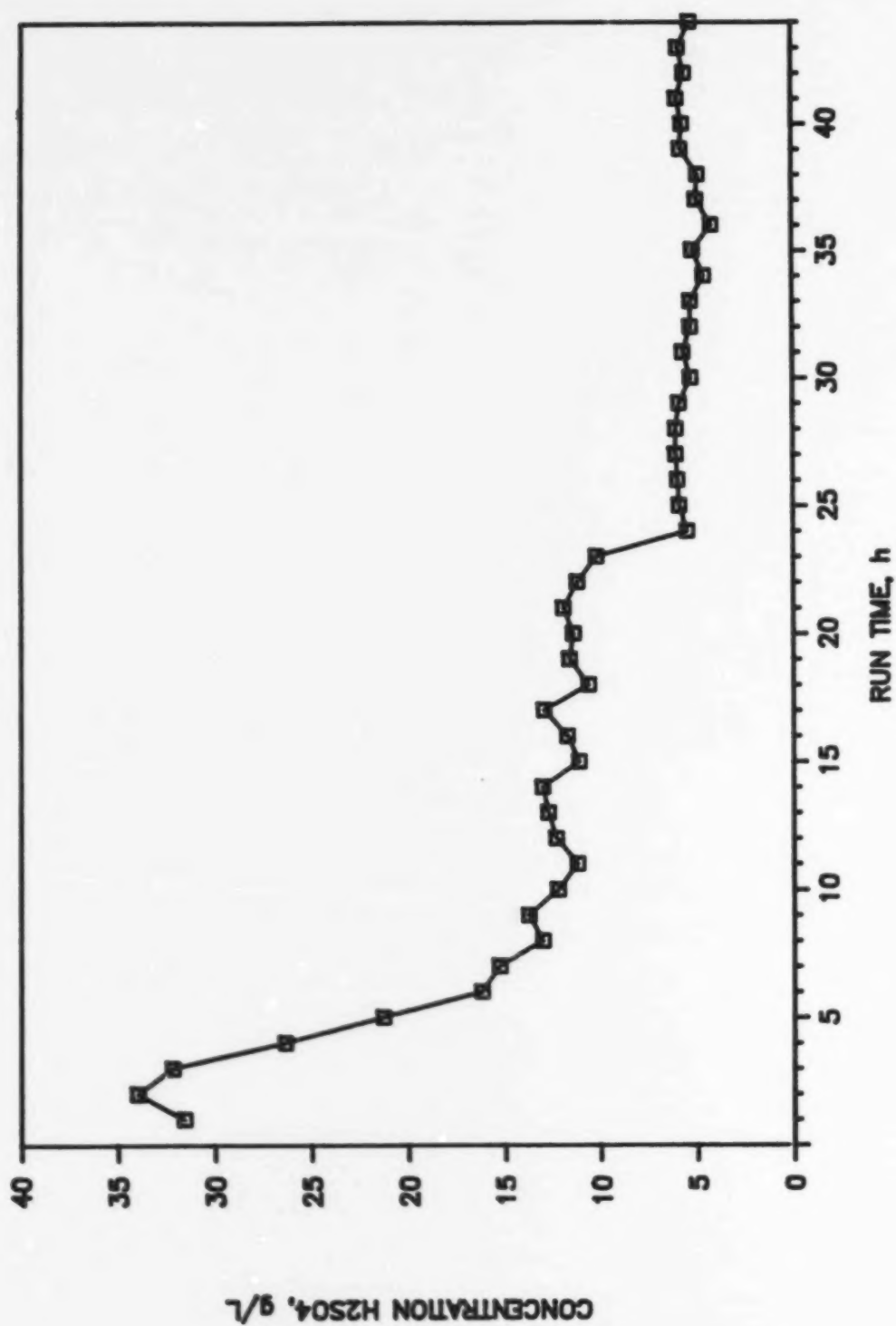


Figure 10.2 Acid In Discharge Solution

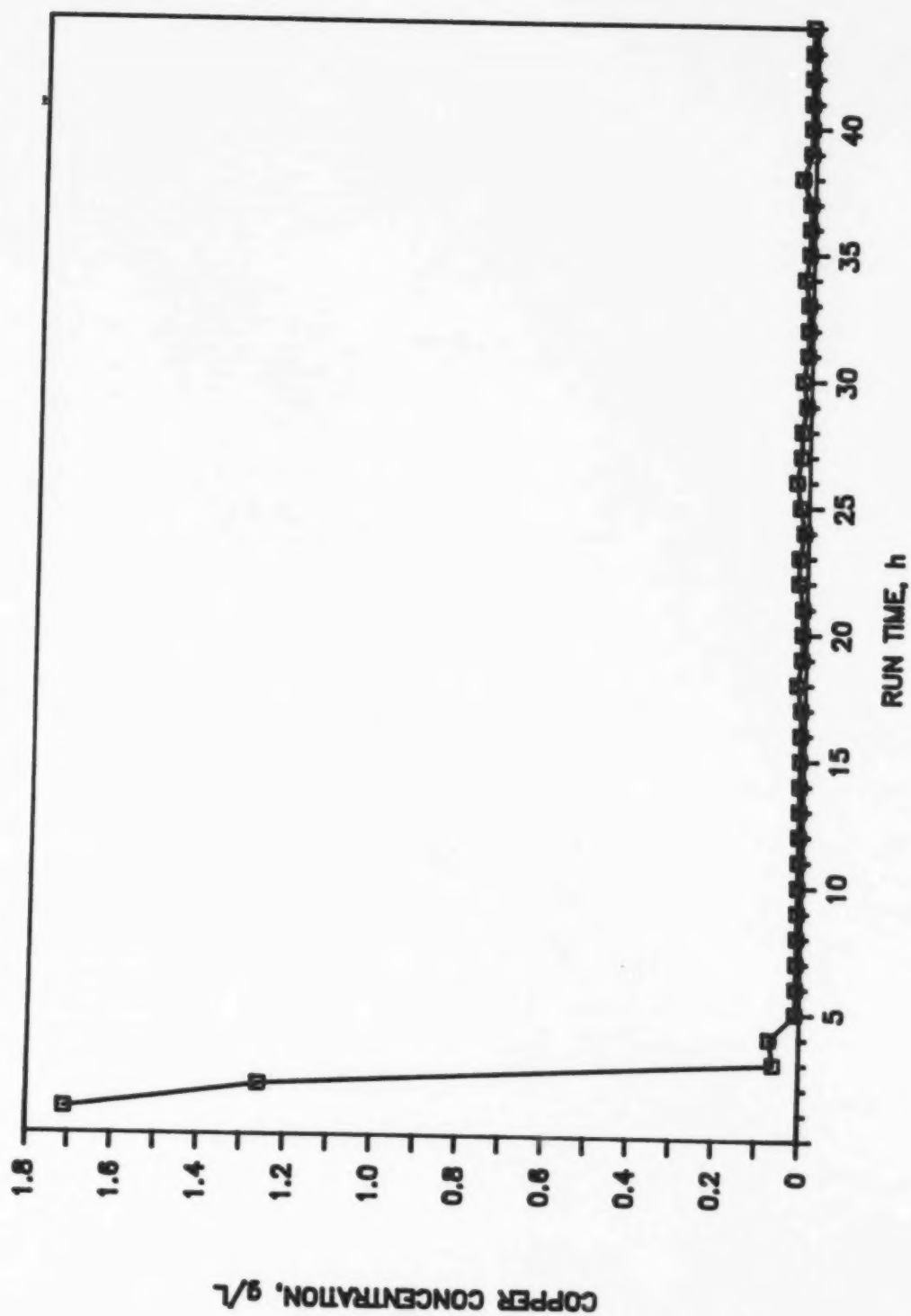


Figure 10.3 Copper In Discharge Solution

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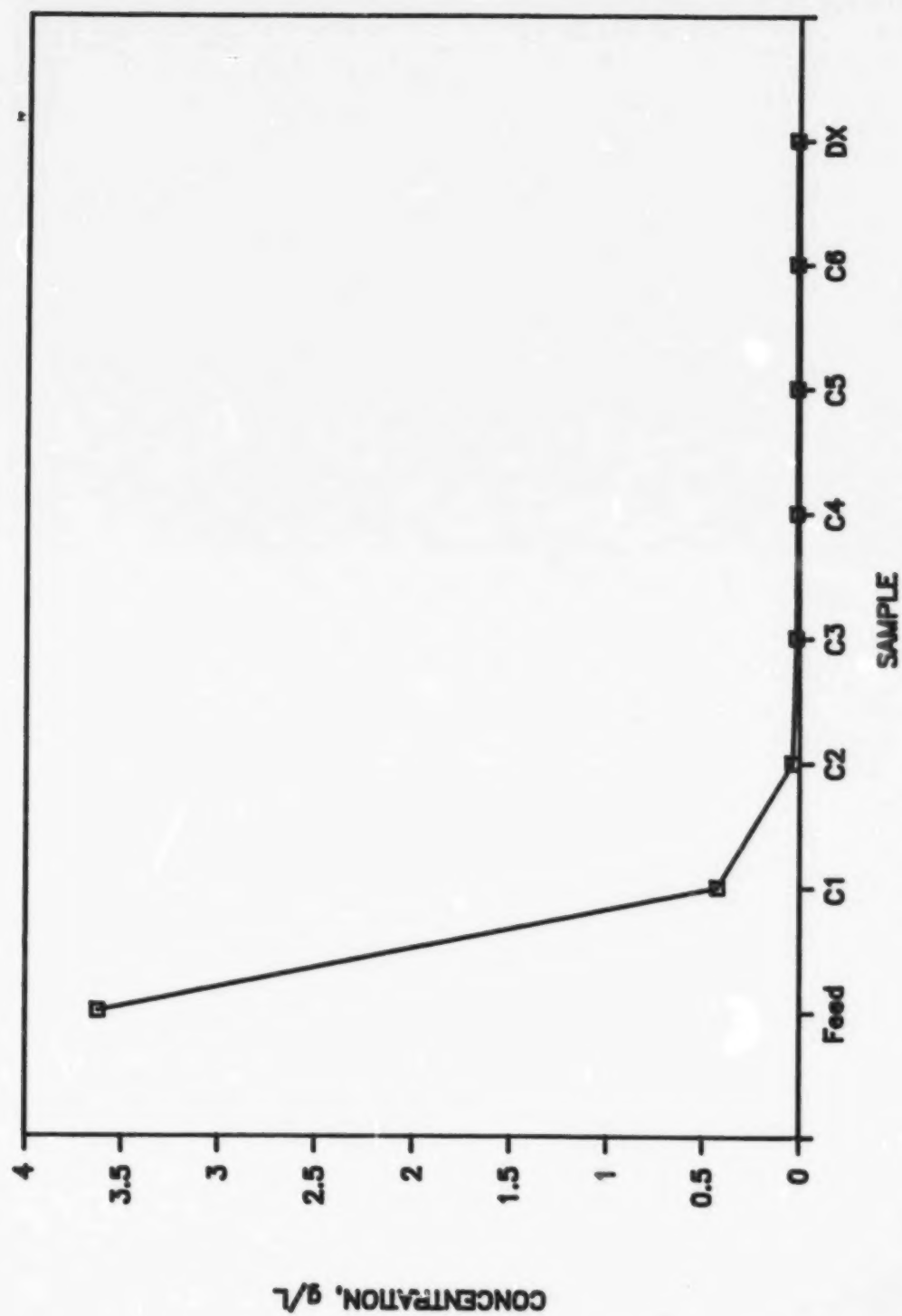


Figure 10.4 Mean Copper Solution Profile

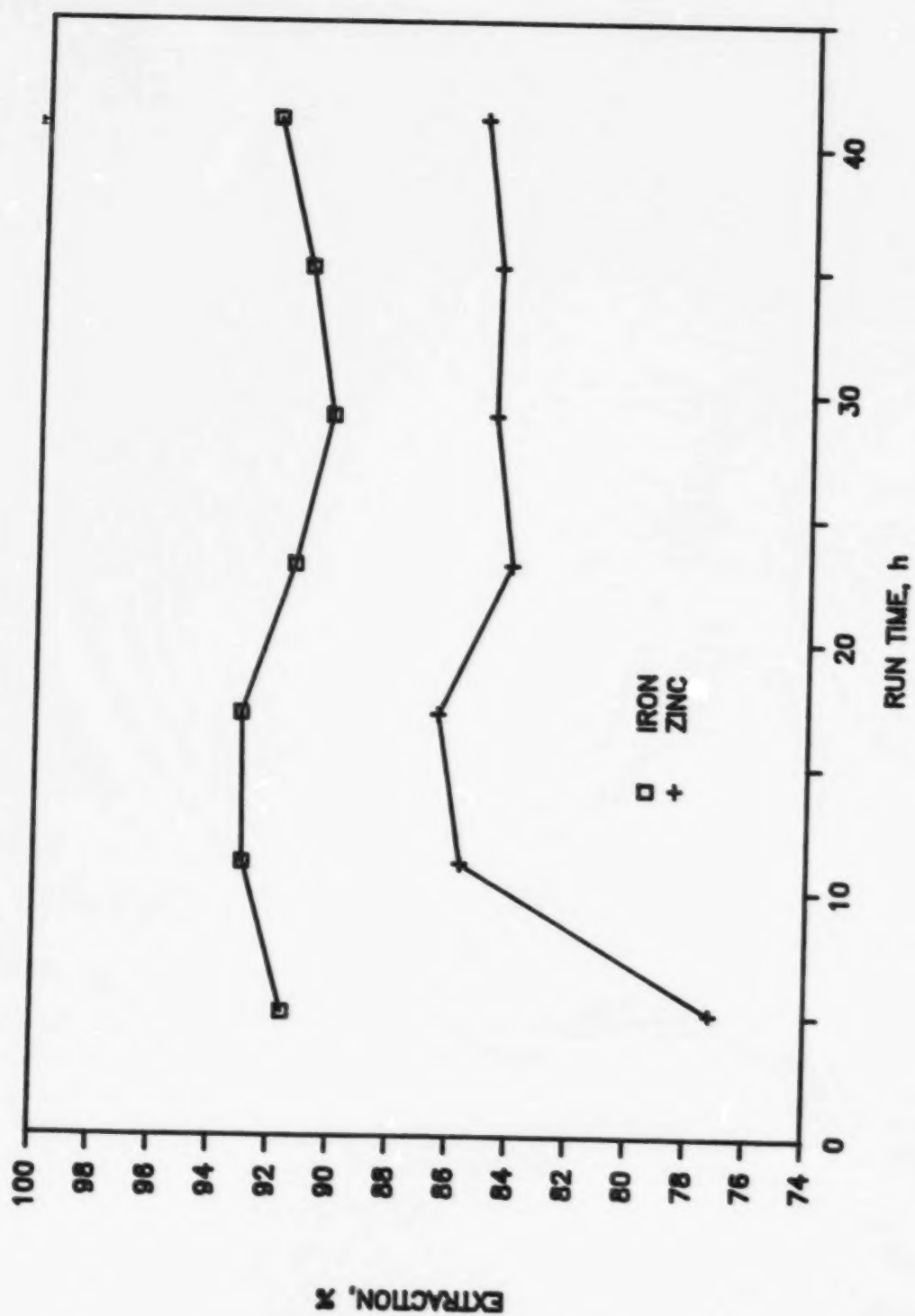


Figure 10.5 Iron and Zinc Extractions in Discharge Solids

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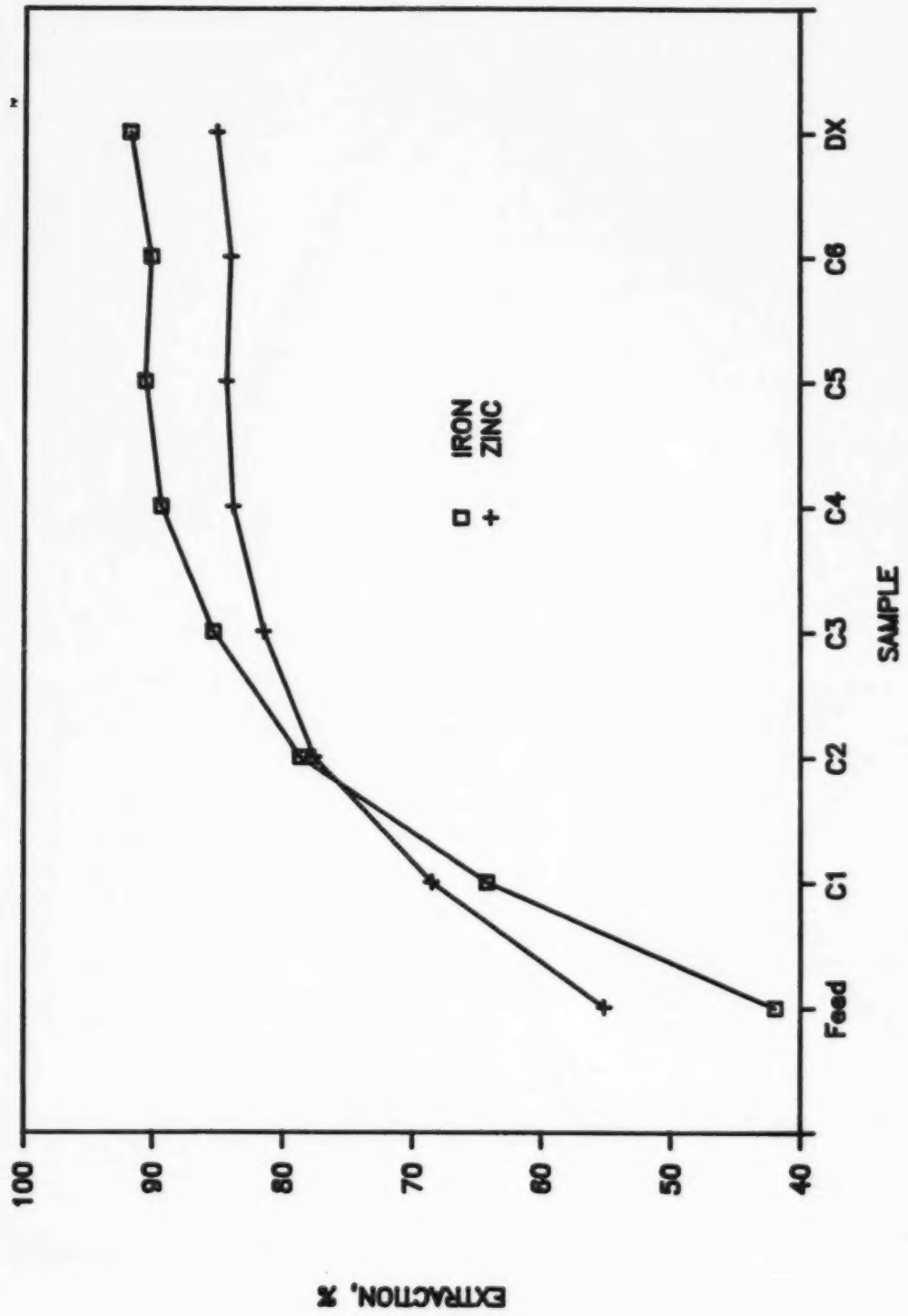


Figure 10.6 Mean Iron and Zinc Extraction Profiles

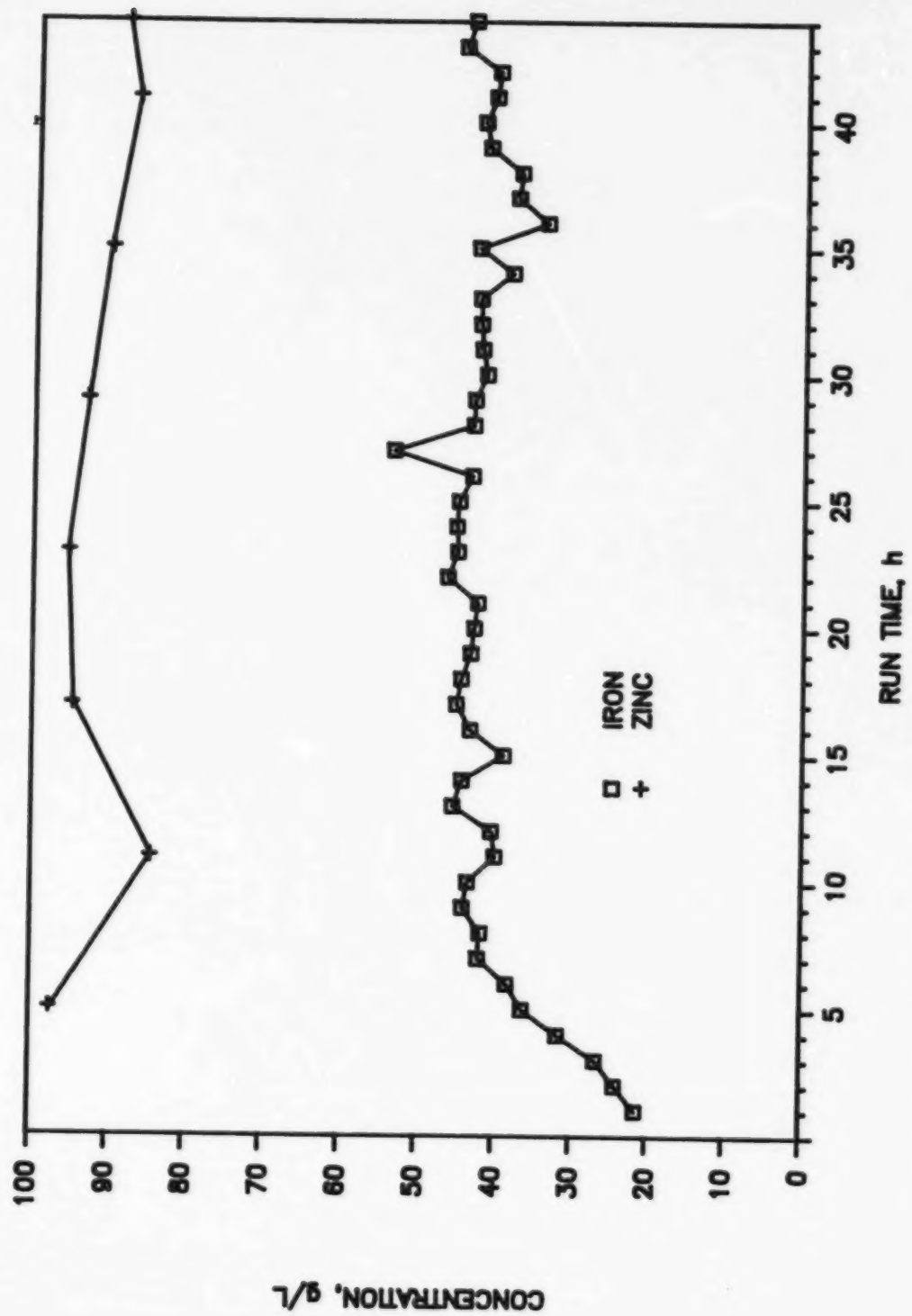


Figure 10.7 Iron and Zinc in Discharge Solution

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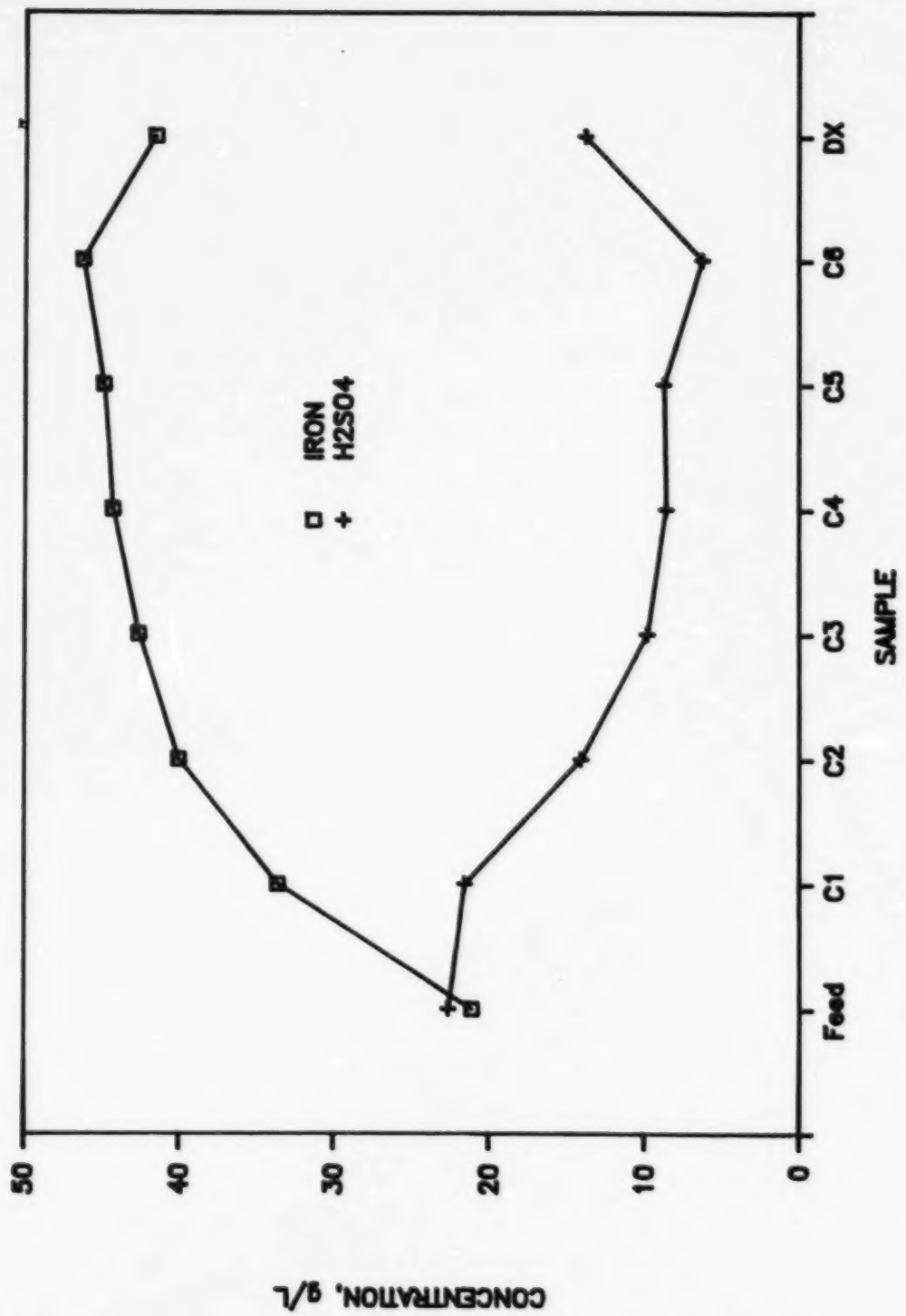


Figure 10.8 Mean Solution Profiles (Periods 1 and 2)

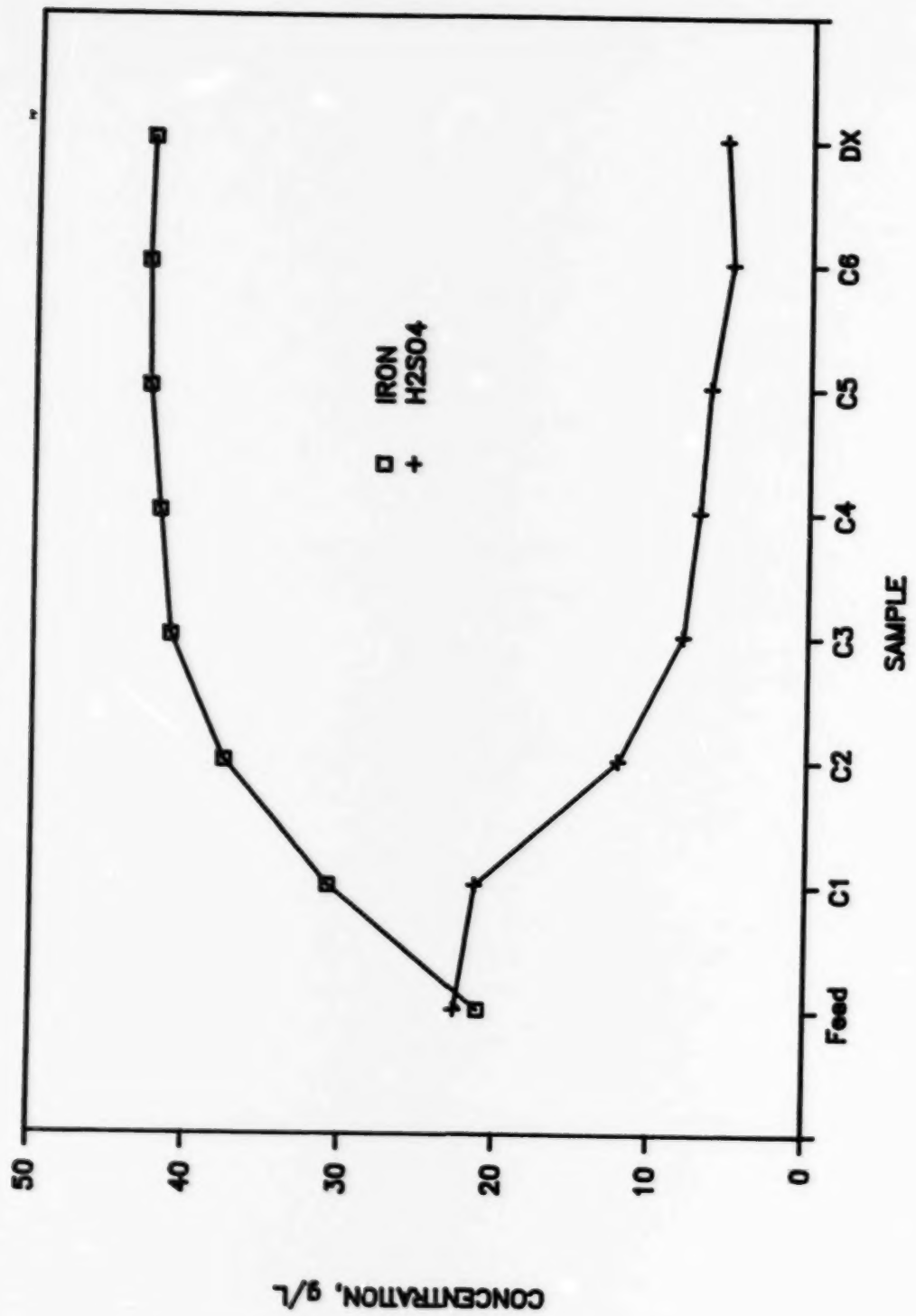


Figure 10.3 Mean Solution Profiles (Periods 3 and 4)

11.0 COPPER FLOTATION

Reducing leach discharge slurry was treated in the copper flotation step. The process objectives were:

to separate a copper sulphide flotation concentrate suitable as feed to a copper smelter; and

to separate the flotation tailing solution from the solids for treatment in iron removal and lead flotation steps, respectively.

Eleven batch flotation tests were conducted prior to the continuous flotation test, including one test on batch reducing leach slurry and ten tests with slurry produced in the continuous reducing leach test. An additional batch flotation test was carried out after completion of the continuous copper flotation test.

The principal purpose of the continuous copper flotation test was to produce feed materials for lead flotation and iron removal testwork. A total of 262 L of reducing leach slurry containing 51 kg of solids was treated in the continuous copper flotation test. The products were 8 kg of cleaner concentrate solids, and 43 kg of scavenger tailing solids.

11.1 BATCH TESTS

The first copper flotation batch test was carried out on batch reducing leach discharge to confirm the viability of the process prior to the continuous testwork (Table 11.1). The results for this batch test are summarized below. Copper recovery was 94% to a high grade concentrate containing 50% Cu.

Fraction	Analysis, %				
	Cd	Cu	Fe	Pb	Zn
Rougher-Scavenger Concentrate	0.02	50.4	0.63	4.62	1.92
Rougher-Scavenger Tailing	0.06	0.22	1.83	22.8	3.08

Fraction	Distribution, %			
	Cu	Fe	Pb	Zn
Rougher-Scavenger Concentrate	94	2	1	4
Rougher-Scavenger Tailing	6	98	99	96

The batch copper flotation tests with continuous reducing leach discharge as the feed are described in Tables 11.2 to 11.11 (Supplementary Tests 1 to 10). These tests were carried out at 60°C, with the slurry diluted to between 1 and 2% solids by volume with

dilute acid solution to simulate recycle of tailing thickener overflow. The results are summarized below.

Test	Reagents	Analysis, %						Copper Recovery %
		First Concentrate			Scavenger Tailing			
		Cu	Pb	Zn	Cu	Pb	Zn	
1	Aero 350	6.4	30.1	3.6	2.6	21.8	3.1	15
2	Aero 350, Z200, Zn(OH) ₂	27.0	5.0	16.8	0.3	22.0	1.4	92
3	Z200	12.3	19.0	7.7	0.2	24.2	1.6	94
4	Z200	19.6	12.8	12.7	0.2	23.8	1.2	95
5	Aero 350, Z200	19.9	15.3	12.5	0.2	25.2	1.3	95
6	Z200, Zn(OH) ₂	17.7	17.6	9.7	1.9	19.8	2.4	41
7	Z200, Gum Arabic	24.1	7.4	15.7	0.2	26.7	1.3	94
8	Aero 350, Z200, Zn(OH) ₂	3.7	41.2	3.1	2.1	16.5	2.2	17
10*	Z200	26.9	7.1	12.8	0.2	21.2	1.2	95

* Cleaner test, with first fraction of concentrate from Test 9 as the feed.

Supplementary Test 1 was carried out under the same conditions as the flotation test with batch reducing leach discharge as the feed, but very poor separation of copper was achieved. It was suspected that the xanthate collector may be more effective at a higher solution pH, and zinc hydroxide sludge was added in the second test to increase the pH to 3.5. A second collector, Z200, was also added. Much better separation of copper was achieved in Test 2, with 92% recovery of copper to the combined rougher-scavenger concentrate.

Although copper recovery was high in Test 2, the grade of the concentrate was low owing to dilution with zinc sulphide. Scavenger flotation was rather inefficient in the continuous zinc flotation step, and the zinc flotation tailing, the feed to the reducing leach, retained 1 to 1.5% Zn as ZnS (see Section 9.3). Zinc sulphide is unattacked in the reducing leach, and is recovered with copper sulphide in copper flotation. Additional batch tests were carried out after completion of the continuous testwork to establish that a high grade copper concentrate could be obtained (see Section 11.3).

Supplementary Tests 3 and 4 were carried out with Z200 alone as the collector, and without pH adjustment. A higher reagent addition rate was used in Test 4, but addition of 360 g/t Z200 in Test 3 appeared to be sufficient. Copper recovery was 94 to 95% in these two tests, slightly higher than in the previous test with Aero 350 added to collect copper sulphide. The first cut of concentrate collected in Test 2 was of higher grade than the corresponding concentrates for Test 3 and 4, but the combined rougher-scavenger

concentrates for the three tests were of similarly poor grade, ranging from 9 to 16% by weight copper. Lead rejection was poor in each case, with 15% of the lead in the feed reporting to the rougher-scavenger concentrate in Test 2; and about 25 % in Tests 3 and 4.

Supplementary Tests 5 and 6 were carried out to confirm that Z200 alone was sufficient for flotation of copper sulphide, and that pH adjustment and addition of Aero 350 were not required. The results for Test 5, with both Aero 350 and Z200 added, were very similar to those of Tests 3 and 4, with Z200 only as the collector. Addition of zinc hydroxide sludge to increase the solution pH in Test 6 resulted in only 41% recovery of copper to the concentrate.

Gum Arabic was added along with Z200 in Test 7 to suppress lead flotation. Copper recovery was 94%, and the first cut of concentrate was of higher grade than in the previous tests with Z200 alone. Separation of copper from lead was still unsatisfactory, however, with 12% of the lead reporting to the combined rougher-scavenger concentrate.

The quality of the concentrate recovered in Tests 3 to 7 was not as high as expected based on the results of Test 2, and the Test 2 conditions were repeated as Test 8. Copper sulphide flotation was very poor in Test 8, with only 17% recovery of copper compared with 92% recovery under identical conditions, but 14 days earlier, in Test 2. It became apparent that aging of the reducing leach slurry was seriously affecting the flotation performance.

Tests 9 and 10 simulated rougher-scavenger and cleaner flotation steps. Test 9 was carried out with Z200 as the collector, as for Test 3. Rougher concentrate from Test 9 was the feed for Test 10. The first cut of concentrate produced in Test 10 contained 27% Cu, and the combined cleaner concentrate contained 12% Cu. Separation of copper from lead was unsatisfactory in all of the tests, even with cleaning, which confirmed that aging of the reducing leach slurry should be minimized prior to flotation.

11.2 CONTINUOUS COPPER FLOTATION

11.2.1 Feed Materials

Discharge slurry from the continuous reducing leach test was the principal feed to the continuous copper flotation test. This slurry was collected in 20 L pails during the reducing leach test. The composition of the solution component of this slurry was variable, particularly with respect to acid concentration. To promote homogeneity of the feed to copper flotation, a 200 L feed tank was used and pails from the beginning and the end of the reducing leach test were added alternately to the feed tank. The average feed composition is given overleaf. The iron in the feed solution is virtually all in the ferrous state, and the acid concentration reported as H_2SO_4 includes sulphuric acid and sulphurous acid which results from dissolution of sulphur dioxide. As solution analyses

were not obtained during copper flotation, the values given are average reducing leach discharge analyses. The solids analyses are average values for the three profiles obtained during continuous copper flotation.

Component	Analysis					
	Cd	Cu	Fe	Pb	H ₂ SO ₄	Zn
Solution, g/L	0.20	0.08	40.9	-	11.0	90.5
Solids, %	0.06	2.85	1.18	22.1	-	2.70

The copper flotation tailing thickener was filled with 60L of synthetic solution prior to the continuous test. The solution was prepared by diluting HBM&S purified neutral leach solution with demineralized water and adding ferrous sulphate and sulphuric acid to appropriate levels. The fill solution analysis is provided below.

Solution	Analysis, g/L			
	Fe ²⁺	Fe	H ₂ SO ₄	Zn
Thickener Fill	33.8	34	5	95

11.2.2 Equipment and Procedures

The continuous copper flotation test circuit was described in detail in Section 6. The equipment flowsheet is provided in Figure 11.1. The four cell flotation unit was divided into one rougher, one cleaner and two scavenger cells. Reducing leach discharge slurry was treated with Z200, 350 g/t, before being pumped to the rougher cell. Rougher concentrate was diluted with overflow from the scavenger tailing thickener and pumped to the cleaner cell. Cleaner concentrate slurry was collected in pails. Scavenger tailing slurry was fed to a 20 cm diameter thickener. A portion of the thickener overflow was recycled to the cleaner cell; the remainder was collected in a drum for feed to the iron removal tests. Thickener underflow was collected in pails for feed to lead flotation.

11.2.3 Operating Parameters

The operating parameters for the continuous flotation test are given in Table 11.12. Samples for flowrate determinations and analysis were collected in an order which least upset the operation. Scavenger tailing, cleaner concentrate, rougher concentrate, scavenger concentrate, feed slurry and feed solution (thickener overflow) streams were collected in the order stated. The feed rate to the circuit was about 0.5 L/min of reducing leach slurry. Percol 351 was added to the scavenger tailing thickener as a 0.5 g/L solution. The addition rate was 11 mL/min, or 150 g/t solids.

11.2.4 Flotation Results

Analyses and inventories for the flotation products are given in Tables 11.13 to 11.15. Average analyses and distributions are summarized in the tables below.

Fraction	Analysis, %					
	Cu	Fe	Pb	S(SO ₄)	S	Zn
Feed	2.85	1.18	22.1	11.9	15.1	2.70
Cleaner Concentrate	16.0	4.15	7.4	4.3	30.5	13.3
Rougher Concentrate	7.34	2.48	19.4	8.4	18.7	7.79
Scavenger Concentrate	2.59	1.25	23.3	11.2	13.7	3.30
Scavenger Tailing	0.43	0.83	25.5	12.3	12.8	1.31

Fraction	Weight kg	Distribution, %					
		Cu	Fe	Pb	S(SO ₄)	S	Zn
Cleaner Concentrate	8	87	47	5	6	30	64
Scavenger Tailing	43*	13	53	95	94	70	36

* Weight of thickener underflow and flotation heel.

The cleaner concentrate contained only 16% Cu on average. Zinc sulphide, elemental sulphur, silica, gypsum and lead sulphate were major diluants, accounting for nearly 60% by weight of the cleaner concentrate. Zinc sulphide and elemental sulphur present in the reducing leach feed would be expected to report to the copper flotation concentrate, and the presence of these minerals in the copper concentrate can best be minimized by efficient operation of the zinc flotation step, as described in Section 11.1. Poor rejection of lead sulphate and gangue minerals would appear to be related to aging of the reducing leach discharge slurry prior to flotation, and better separation is expected if aging is minimized.

The continuous scavenger tailing solids contained 0.43% Cu by weight, compared with about 0.2% Cu in many of the batch tests. Overall recovery of copper to the concentrate was correspondingly lower in the continuous test, 87%, compared with about 95% in the batch tests.

The continuous scavenger tailing contained 1.3% Zn and 25.5% Pb. Using a lead tie, this represented a net zinc extraction for the process of 94%.

11.2.5 Thickener Operation

The addition rate of flocculant (Percol 351) to the copper flotation scavenger tailing thickener was 150 g/t scavenger tailing solids. The solids settled slowly and the average pulp density of the underflow was 1543 g/L (18% solids).

11.3 ADDITIONAL BATCH TESTS

The grade of the copper concentrate obtained in the continuous copper flotation test was very low, and additional batch tests were conducted to determine if a higher grade concentrate could be obtained. The low grade of the concentrate in continuous flotation was a result of inefficient flotation of zinc sulphide and elemental sulphur in the continuous zinc flotation test, and poor rejection of lead sulphate and gangue minerals in the continuous copper flotation test due to excessive aging of the feed slurry.

Slurry from the continuous neutralization-reduction test was treated to separate residual zinc sulphide and elemental sulphur in batch flotation tests. The results for these tests are described in Section 9.3. The zinc flotation tailing solids contained about 6% Zn, compared with about 7.5% Zn for the continuous test. The batch tailing slurry was treated in a reducing leach, under the same conditions as Test 2, Table 10.2. The zinc flotation tailing slurry, 2.5 L, was mixed with 10 g of elemental sulphur, under 100 kPa sulphur dioxide overpressure, at 100 °C for four hours. Upon completion of the leach test, the slurry was diluted with warm sulphuric acid solution and immediately subjected to flotation. Both rougher-scavenger and cleaner flotation rate tests were carried out, as described in Tables 11.16 and 11.17, and summarized below.

Fraction	Analysis, %				
	Cd	Cu	Fe	Pb	Zn
Cleaner Concentrate	0.01	49.0	0.7	0.2	1.14
Rougher-Scavenger Concentrate	0.04	36.4	1.45	11.1	3.46
Rougher-Scavenger Tailing	0.05	0.3	2.17	18.3	1.53

Fraction	Distribution, %			
	Cu	Fe	Pb	Zn
Cleaner Concentrate	87.9	1.5	0.1	3.8
Cleaner Tailing	0.7	2.6	3.7	8.8
Rougher-Scavenger Tailing	11.4	95.9	96.2	87.3

Improved flotation of zinc in the batch zinc flotation tests allowed for good separation of copper and zinc to their respective flotation concentrates. The copper flotation concentrate contained 49% Cu and only 1.1% zinc. Copper recovery was 88%.

Flotation of the reducing leach slurry immediately after discharge from the autoclave likely also contributed to the production of a high grade copper concentrate. The cleaner concentrate contained less than 1% by weight Pb, and only 0.1% of the lead in the feed to flotation. Gangue minerals such as silica and gypsum were also successfully rejected, with only 0.3% SiO₂ and 0.01% Ca in the cleaner concentrate.

11.4 CONCLUSIONS AND RECOMMENDATIONS

The reducing leach residue is amenable to flotation to separate a copper concentrate.

Although the reducing leach solids contain some elemental sulphur, which aids in the flotation of copper sulphide, addition of a collecting agent such as Z200 is recommended.

A high grade copper concentrate can only be obtained if zinc sulphide is effectively removed from the process slurry in the previous zinc flotation step, and if the reducing leach slurry is not allowed to age for an excessive period of time. If the above two conditions are met, 85 to 95% recovery of copper to a concentrate containing about 50% by weight copper can be attained.

Table 11.1 Batch Copper Flotation: Test 1

Feed Material	: 1.42 L discharge slurry from reducing leach Test 2 diluted to 2.0 L with 10 g/L H ₂ SO ₄ solution
Solids Concentration	: 72 g/L slurry (2% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (10 g/L)
Temperature	: 75°C
Reagents	: Aero 350, 175 g/t
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (275 g/t)

Solids	Calculated Head	Concentrate	Tailing
Time, min	-	2	-
Weight, g	143.3	9.2	134.1
Analysis, %			
As	0.71	0.07	0.25
Cd	0.06	0.02	0.06
Ca	9.84	0.16	10.5
Cu	3.44	50.4	0.22
Fe	1.75	0.63	1.83
Pb	21.6	4.62	22.8
SiO ₂	7.49	1.20	7.92
Zn	3.01	1.92	3.08
Distribution, %			
Cu	100	94	6
Fe	100	2	98
Pb	100	1	99
Zn	100	4	96

Table 11.2 Batch Copper Flotation: Supplementary Test 1

Date	: July 26, 1989
Feed Material	: 0.5 L continuous reducing leach discharge slurry collected at 34 h run time, diluted with filtrate.
Solids Concentration	: 42 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 60°C
Reagents	: Aero 350, 150 g/t
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	2	5	10	-
Weight, g	84.3	2.4	1.7	3.1	77.1
Analysis, %					
As	0.26	0.48	0.51	0.59	0.23
Cd	0.06	0.08	0.08	0.09	0.06
Ca	9.46	3.15	3.61	1.68	10.1
Cu	2.79	6.45	4.75	3.98	2.59
Fe	2.31	3.33	3.27	3.55	2.21
Pb	22.9	30.1	32.7	38.1	21.8
SiO ₂	5.90	12.7	9.51	8.79	5.49
Zn	3.15	3.63	3.35	3.56	3.11
Distribution, %					
Cu	100	6.6	3.4	5.2	84.8
Fe	100	4.1	2.8	5.6	87.4
Pb	100	3.8	2.9	6.1	87.2
Zn	100	3.3	2.2	4.2	90.4

Table 11.3 Batch Copper Flotation: Supplementary Test 2

Date	: July 27, 1989
Feed Material	: 0.67 L continuous reducing leach discharge slurry collected between 42 and 44 h run time; 1.33 L filtrate
Solids Concentration	: 65 g/L slurry (2% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: 3.5 (adjusted with zinc hydroxide sludge)
Temperature	: 60°C
Reagents	: Aero 350, 100 g/t; Z200, 325 g/t
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Tailing
Time, min	-	2	8	-
Weight, g	129.4	10.5	10.4	108.5
Analysis, %				
As	0.43	0.17	0.62	0.44
Cd	0.06	0.08	0.09	0.06
Ca	12.0	0.05	6.45	13.7
Cu	2.90	27.0	5.75	0.29
Fe	1.89	5.88	2.41	1.45
Pb	21.2	5.04	29.5	22.0
SiO ₂	6.48	0.92	7.29	6.94
Zn	2.92	16.8	5.20	1.36
Distribution, %				
Cu	100	75.6	16.0	8.4
Fe	100	25.3	10.3	64.4
Pb	100	1.9	11.2	86.9
Zn	100	46.7	14.3	39.0

Table 11.4 Batch Copper Flotation: Supplementary Test 3

Date	: July 27, 1989
Feed Material	: 0.67 L continuous reducing leach discharge slurry collected between 42 and 44 h run time; 1.33 L filtrate
Solids Concentration	: 58 g/L slurry (1.5% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 60°C
Reagents	: Z200, 360 g/t
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	2	5	10	20	-
Weight, g	116.3	8.8	7.8	7.3	8.8	83.6
Analysis, %						
As	0.17	0.20	0.19	0.24	0.23	0.15
Cd	0.06	0.07	0.08	0.08	0.07	0.06
Ca	11.8	6.65	5.12	6.66	9.92	13.6
Cu	2.75	12.3	13.8	9.09	2.13	0.23
Fe	1.65	3.84	3.44	2.12	1.37	1.24
Pb	23.8	19.0	19.5	23.9	29.2	24.2
SiO ₂	6.74	5.14	5.61	7.63	6.86	6.92
Zn	3.01	7.70	9.43	6.93	2.87	1.59
Distribution, %						
Cu	100	33.8	33.6	20.7	5.8	6.0
Fe	100	17.6	14.0	8.1	6.3	54.0
Pb	100	6.0	5.5	6.3	9.3	72.9
Zn	100	19.4	21.0	14.4	7.2	38.0

Table 11.5 Batch Copper Flotation: Supplementary Test 4

Date : July 31, 1989
 Feed Material : 0.67 L continuous reducing leach discharge slurry collected between 42 and 44 h run time; 1.33 L filtrate
 Solids Concentration : 58 g/L slurry (1.5% by volume)
 Slurry Volume : 2.0 L
 Solution pH (g/L H₂SO₄) : (5 g/L)
 Temperature : 60°C
 Reagents : Z200, 900 g/t
 Conditioning Time : 1 minute
 Frothing Agent : none

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	2	5	10	-
Weight, g	115.4	13.1	5.2	8.9	88.2
Analysis, %					
As	0.25	0.17	0.34	0.35	0.25
Cd	0.06	0.07	0.09	0.07	0.05
Ca	11.2	1.83	1.88	7.87	13.5
Cu	2.80	19.6	7.11	1.40	0.19
Fe	1.41	4.51	2.48	1.12	0.92
Pb	23.6	12.8	30.8	32.7	23.8
SiO ₂	7.06	5.55	13.2	10.4	6.58
S(SO ₄)	12.8	3.71	6.46	11.5	14.6
S	16.5	29.5	15.1	12.9	15.0
Zn	2.96	12.7	8.76	2.30	1.24
Distribution, %					
Cu	100	79.5	11.4	3.9	5.2
Fe	100	36.2	7.9	6.1	49.8
Pb	100	6.2	5.9	10.7	77.2
Zn	100	48.7	13.3	6.0	32.0

Table 11.6 Batch Copper Flotation: Supplementary Test 5

Date	: July 31, 1989
Feed Materials	: 0.67 L continuous reducing leach discharge slurry collected between 42 and 44 h run time; 1.33 L filtrate
Solids Concentration	: 54 g/L slurry (1.4% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 60°C
Reagents	: Aero 350, 125 g/t; Z200, 385 g/t
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	5	10	-
Weight, g	109.0	11.1	2.9	3.4	8.7	82.9
Analyses, %						
As	0.30	0.25	0.28	0.40	0.38	0.30
Cd	0.06	0.08	0.09	0.09	0.07	0.06
Ca	11.1	1.98	0.30	1.60	7.43	13.5
Cu	2.99	19.9	19.2	8.28	0.72	0.18
Fe	1.44	4.50	4.12	2.35	1.05	0.94
Pb	24.9	15.3	17.5	31.8	33.7	25.2
SiO ₂	7.51	5.01	7.09	13.9	12.2	7.11
S(SO ₄)	12.5	4.09	3.14	6.26	11.3	14.4
S	16.3	29.3	24.7	14.9	12.2	14.8
Zn	3.12	12.5	15.1	7.53	2.46	1.33
Distribution, %						
Cu	100	67.8	17.1	8.6	1.9	4.6
Fe	100	31.8	7.6	5.1	5.8	49.6
Pb	100	6.3	1.9	4.0	10.8	77.1
Zn	100	40.8	12.9	7.5	6.3	32.4

Table 11.7 Batch Copper Flotation: Supplementary Test 6

Date	: July 31, 1989
Feed Material	: 0.67 L continuous reducing leach discharge slurry collected between 42 and 44 h run time; 1.33 L filtrate
Solids Concentration	: 65 g/L slurry (1.6% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: 3.5 (adjusted with zinc hydroxide sludge)
Temperature	: 60°C
Reagents	: Z200, 320 g/t
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	2	5	10	15	-
Weight, g	129.9	5.8	5.8	13.9	1.6	102.8
Analysis, %						
As	0.50	0.30	0.60	0.62	0.99	0.48
Cd	0.06	0.07	0.07	0.06	0.08	0.06
Ca	12.3	2.34	8.54	11.4	5.55	13.3
Cu	2.56	17.7	2.32	1.28	1.79	1.90
Fe	1.70	4.69	1.73	1.44	2.53	1.55
Pb	20.9	17.6	30.4	24.9	30.7	19.8
SiO ₂	6.73	4.01	7.48	6.92	13.3	6.71
S(SO ₄)	12.4	4.90	11.5	4.86	25.2	13.7
S	n.a.	28.1	13.9	14.3	n.a.	43.6
Zn	2.67	9.72	2.40	1.66	2.38	2.43
Distribution, %						
Cu	100	30.9	4.0	5.4	0.9	58.8
Fe	100	12.3	4.6	9.1	1.8	72.2
Pb	100	3.8	6.5	12.8	1.8	75.1
Zn	100	16.2	4.0	6.6	1.1	72.0

n.a. = not analyzed

Table 11.8 Batch Copper Flotation: Supplementary Test 7

Date	: August 8, 1989
Feed Material	: 0.67 L continuous reducing leach discharge slurry collected between 42 and 44 h run time; 1.33 L filtrate
Solids Concentration	: 39 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 60°C
Reagents	: Z200, 540 g/t; Gum Arabic*, 130 g/t
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	2	5	10	20	-
Weight, g	77.3	5.1	3.7	2.0	7.0	59.5
Analysis, %						
As	0.18	0.58	1.03	1.15	0.34	0.04
Cd	0.06	0.09	0.09	0.07	0.04	0.06
Ca	10.2	0.04	0.15	4.07	13.9	11.5
Cu	2.75	24.1	12.9	7.90	1.81	0.23
Fe	1.25	5.34	2.94	2.13	1.04	0.79
Pb	23.4	7.38	23.7	17.2	8.94	26.7
SiO ₂	6.89	4.84	15.1	23.1	13.0	5.29
S(SO ₄)	11.5	1.35	3.85	5.83	12.3	13.0
S	15.8	35.8	19.2	13.2	15.1	14.0
Zn	2.78	15.7	9.12	4.68	2.42	1.26
Distribution, %						
Cu	100	57.8	22.4	7.4	6.0	6.4
Fe	100	28.2	11.2	4.4	7.5	48.6
Pb	100	2.1	4.8	1.9	3.5	87.7
Zn	100	37.2	15.7	4.4	7.9	34.9

* added during heat up

Table 11.9 Batch Copper Flotation: Supplementary Test 8

Date	: August 8, 1989
Feed Material	: 0.67 L continuous reducing leach discharge slurry collected between 42 and 44 h run time; 1.33 L filtrate
Solids Concentration	: 52 g/L slurry (1.3% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: 3.5 (adjusted with zinc hydroxide sludge)
Temperature	: 60°C
Reagents	: Aero 350, 135 g/t; Z200, 400 g/t*
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	2	5	10	15	-
Weight, g	103.6	1.4	2.6	6.6	6.3	86.7
Analysis, %						
As	1.16	2.23	2.30	1.52	1.10	1.08
Cd	0.05	0.11	0.10	0.06	0.04	0.05
Ca	12.4	0.30	3.78	13.0	17.2	12.4
Cu	2.10	3.68	4.33	2.55	0.68	2.08
Fe	1.83	3.04	2.87	1.75	1.26	1.83
Pb	16.9	41.2	33.7	17.0	10.3	16.5
SiO ₂	4.69	10.1	8.10	4.11	2.98	4.67
S(SO ₄)	12.5	6.61	8.21	13.0	15.3	12.5
S	15.6	10.1	12.2	15.8	16.6	15.7
Zn	2.08	3.13	2.65	1.51	0.82	2.18
Distribution, %						
Cu	100	2.4	5.2	7.7	2.0	82.8
Fe	100	2.2	3.9	6.1	4.2	83.6
Pb	100	3.3	5.0	6.4	3.7	81.6
Zn	100	2.0	3.2	4.6	2.4	87.7

* Aero 350 added initially, but no concentrate produced upon aeration. Z200 was then added and the test continued.

Table 11.10 Batch Copper Flotation: Supplementary Test 9

Date	: August 8, 1989
Feed Material	: 4.0 L continuous reducing leach discharge slurry collected between 42 and 44 h run time
Solids Concentration	: 173 g/L slurry (4% by volume)
Slurry Volume	: 4.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 60°C
Reagents	: Z200, 365 g/t
Conditioning Time	: 1 minute
Frothing Agent	: none

Solids	Calc Head*	Con 2*	Con 3	Con 4	Con 5	Tailing
Time, min	-	5	10	20	30	-
Weight, g	691.2	68.3	37.9	39.6	43.9	376.0
Analysis, %						
As	0.16	0.28	0.34	0.22	0.12	0.06
Cd	0.05	0.06	0.07	0.07	0.06	0.04
Ca	11.4	8.34	6.15	7.59	9.21	14.4
Cu	2.72	6.62	9.45	4.89	2.03	0.23
Fe	1.21	1.89	2.37	1.61	1.31	0.72
Pb	22.6	23.7	22.2	24.6	22.8	21.2
SiO ₂	6.44	7.56	8.85	10.3	9.24	4.93
S(SO ₄)	12.2	9.67	8.40	9.88	9.80	14.1
S	16.3	18.4	19.9	16.6	15.8	15.3
Zn	3.60	4.31	6.16	4.98	5.17	1.19
Distribution, %						
Cu	100	24.0	19.0	10.3	4.7	4.6
Fe	100	15.4	10.8	7.6	6.9	32.4
Pb	100	10.4	5.4	6.2	6.4	51.1
Zn	100	11.8	9.4	7.9	9.1	18.0

* 2 minute concentrate slurry saved for cleaner flotation, Test 10

Table 11.11 Batch Copper Flotation: Supplementary Test 10 (Cleaner)

Date : August 8, 1989
 Feed Material : 0.8 L concentrate slurry from Test 9, 1.2 L filtrate
 Solids Concentration : 63 g/L slurry (1.6% by volume)
 Slurry Volume : 2.0 L
 Solution pH (g/L H₂SO₄) : (5 g/L)
 Temperature : 60°C
 Reagents : none
 Frothing Agent : none

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	2	5	10	20	-
Weight, g	125.5	15.2	7.7	16.2	16.6	69.8
Analysis, %						
As	0.31	0.93	0.99	0.45	0.22	0.09
Cd	0.07	0.08	0.09	0.07	0.06	0.06
Ca	7.88	0.35	0.39	4.84	9.79	10.6
Cu	5.59	26.9	20.5	5.35	1.84	0.26
Fe	1.79	5.80	4.02	1.64	1.08	0.87
Pb	25.4	7.13	14.0	23.3	24.2	31.4
SiO ₂	7.45	4.37	9.62	11.4	11.9	5.91
S(SO ₄)	10.5	1.67	2.90	7.53	11.8	13.6
S	17.4	35.7	27.3	13.8	13.7	14.1
Zn	8.66	12.8	12.0	4.62	2.53	9.79
Distribution, %						
Cu	100	58.2	22.5	12.3	4.4	2.6
Fe	100	39.3	13.8	11.8	8.0	27.1
Pb	100	3.4	3.4	11.8	12.6	68.8
Zn	100	17.9	8.5	6.9	3.9	62.9

Table 11.12 Copper Flotation Continuous Operating Parameters

Run Time h	Sample	Rate mL/min	Pulp Density g/L	Solids *		Retention Time	
				Wt %	Vol %	Cell	Min
2	Feed Slurry	490	1485	12	4		
	Cleaner Con.	416	1425	9	3	Cleaner	22.0
	Rougher Con.	588	1480	8	3	Rougher	5.2
	Scav. Con.	3420	1416	6	2	Scav.	12.0
	Scav. Tailing	394	1439	8	3		
	Feed Solution	504	1356	-	-		
3	Feed Slurry	496	1481	12	4		
	Cleaner Con.	290	1396	8	3	Cleaner	18.3
	Rougher Con.	803	1421	6	2	Rougher	10.2
	Scav. Con.	834	1407	5	2	Scav.	31.0
	Scav. Tailing	564	1425	7	2		
	Feed Solution	507	1356	-	-		
4	Feed Slurry	430	1480	12	4		
	Cleaner Con.	187	1371	4	2	Cleaner	22.5
	Rougher Con.	628	1414	6	2	Rougher	3.9
	Scav. Con.	4860	1406	5	2	Scav.	8.7
	Scav. Tailing	634	1426	8	3		
	Feed Solution	440	1358	-	-		
5	Cleaner Con.	250	1365	3	1		
	Scav. Tailing	645	1428	7	3		

* Based on weights of dry solids collected

Table 11.13 Continuous Copper Flotation Analyses (As to Fe)

Profile	Stream	Analysis, %				
		As	Cd	Ca	Cu	Fe
1 (2h)	Feed	0.12	0.06	11.1	2.90	1.24
	Cleaner Concentrate	0.83	0.10	3.12	17.5	4.87
	Rougher Concentrate	0.47	0.08	6.23	6.52	2.42
	Scavenger Concentrate	0.30	0.06	9.61	2.57	1.31
	Scavenger Tailing	0.10	0.05	11.0	0.42	0.81
	Thickener Underflow	<0.02	0.06	11.8	0.59	1.00
2 (3h)	Feed	0.03	0.06	10.7	2.90	1.15
	Cleaner Concentrate	1.03	0.09	3.75	17.3	5.05
	Rougher Concentrate	0.40	0.07	7.50	7.15	2.19
	Scavenger Concentrate	0.41	0.06	9.50	3.70	1.34
	Scavenger Tailing	0.11	0.06	10.4	0.46	0.86
	Thickener Underflow	<0.02	0.05	11.5	0.54	0.89
3 (4h)	Feed	0.05	0.06	10.9	2.75	1.16
	Cleaner Concentrate	0.83	0.07	4.30	16.1	3.57
	Rougher Concentrate	0.32	0.07	6.52	8.35	2.82
	Scavenger Concentrate	0.14	0.06	9.99	1.49	1.09
	Scavenger Tailing	<0.02	0.06	11.6	0.44	0.86
	Thickener Underflow	<0.02	0.05	11.4	0.51	0.88
4 (5h)	Cleaner Concentrate	1.18	0.08	4.04	13.2	3.10
	Scavenger Tailing	0.14	0.05	11.7	0.40	0.80
Average	Feed	0.07	0.06	10.9	2.85	1.18
	Cleaner Concentrate	0.97	0.09	3.80	16.0	4.15
	Rougher Concentrate	0.40	0.07	6.75	7.34	2.48
	Scavenger Concentrate	0.28	0.06	9.70	2.59	1.25
	Scavenger Tailing	0.09	0.06	11.2	0.43	0.83
	Thickener Underflow	0.02	0.05	11.6	0.55	0.92

Table 11.14 Continuous Copper Flotation Analyses (Pb to Zn)

Profile	Stream	Analysis, %				
		Pb	SiO ₂	S(SO ₄)	S	Zn
1	Feed	22.4	7.49	11.9	15.3	2.77
	Cleaner Concentrate	4.9	7.12	3.2	31.6	17.8
	Rougher Concentrate	21.2	8.94	8.4	16.8	8.10
	Scavenger Concentrate	23.5	8.19	11.1	14.0	3.55
	Scavenger Tailing	24.2	7.17	12.6	13.2	1.26
	Thickener Underflow	24.4	7.55	12.8	12.9	1.53
2	Feed	21.9	7.27	11.9	14.9	2.66
	Cleaner Concentrate	6.1	7.17	3.9	30.2	15.1
	Rougher Concentrate	18.6	8.17	9.0	19.2	7.03
	Scavenger Concentrate	20.1	9.37	10.8	13.5	4.05
	Scavenger Tailing	27.6	7.70	10.8	12.7	1.43
	Thickener Underflow	24.4	7.36	12.9	12.7	1.42
3	Feed	22.0	7.34	12.0	15.2	2.67
	Cleaner Concentrate	6.9	11.3	4.9	26.5	10.9
	Rougher Concentrate	18.4	8.79	7.9	20.1	8.25
	Scavenger Concentrate	26.3	8.30	11.7	13.6	2.31
	Scavenger Tailing	25.3	7.49	12.8	12.4	1.29
	Thickener Underflow	24.8	7.40	12.9	12.6	1.40
4	Cleaner Concentrate	11.8	12.7	5.2	33.6	9.28
	Scavenger Tailing	24.8	7.17	12.8	12.9	1.25
Average	Feed	22.1	7.37	11.9	15.1	2.70
	Cleaner Concentrate	7.4	9.56	4.3	30.5	13.3
	Rougher Concentrate	19.4	8.64	8.4	18.7	7.79
	Scavenger Concentrate	23.3	8.62	11.2	13.7	3.30
	Scavenger Tailing	25.5	7.38	12.3	12.8	1.31
	Thickener Underflow	24.5	7.44	12.9	12.7	1.45

Table 11.15 Continuous Copper Flotation Product Inventory

Cleaner Concentrate		
Pail Number	Slurry Weight, kg	Slurry P.D., g/L
1	26.62	1491
2	19.92	1454
3	17.80	1465

Total 64.34 kg slurry
 7.69 kg solids

Scavenger Tailing Thickener Underflow		
Pail Number	Slurry Weight, kg	Slurry P.D., g/L
1	24.33	1575
2	23.49	1530
3	21.89	1563
4	22.90	1596
5	22.92	1667

Total 115.53 kg slurry
 25.35 kg solids

Flotation Heel *		
Pail Number	Slurry Weight, kg	Slurry P.D., g/L
1	7.94	1470
2	23.94	1482
3	25.24	1476
4	27.17	1453
5	24.19	1470
6	22.42	1446
7	26.04	1459

Total 156.94 kg slurry
 17.61 kg solids

* Similar in composition to scavenger tailing

Table 11.16 Batch Copper Flotation: Supplementary Test 11

Feed Material	: Reducing leach slurry from treatment of zinc flotation tailing, Supplementary Batch Tests 8 to 10
Solids Concentration	: 45 g/L slurry (1.1% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 60°C
Reagents	: Z200, 460 g/t
Conditioning Time	: 1 minute
Frothing Agent	: Dowiroth 200, 1 drop (230 g/t)

Solids	Calc Head	Con 1	Con 2	Tailing
Time, min	-	5	10	-
Weight, g	91.1	4.3	2	84.8
Analysis, %				
As	0.34	0.06	0.56	0.35
Cd	0.05	0.01	0.08	0.06
Ca	11.8	0.01	0.30	12.7
Cu	2.83	45.9	13.0	0.40
Fe	2.34	0.90	3.08	2.39
Pb	21.1	5.78	35.2	21.5
SiO ₂	7.85	1.07	7.4	8.2
S°	n.a.	20.3	n.a.	0.7
Zn	1.78	1.32	3.68	1.76
Distribution, %				
As	100	0.8	3.6	95.6
Cd	100	1.1	3.1	95.7
Ca	100	0.0	0.1	99.9
Cu	100	76.6	10.1	13.3
Fe	100	1.8	2.9	95.3
Pb	100	1.3	3.7	95.0
SiO ₂	100	0.6	2.1	97.3
Zn	100	3.5	4.5	92.0

n.a. = not analyzed

Table 11.17 Batch Copper Flotation: Supplementary Test 12 (Cleaner)

Feed Material	: Rougher-scavenger concentrate (0 to 10 min) produced as in Test 11, diluted with 5 g/L H ₂ SO ₄ solution
Solids Concentration	: 8 g/L slurry (0.2% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 60°C
Reagents	: Z200, 390 g/t, used for rougher-scavenger only
Conditioning Time	: 1 minute
Frothing Agent	: Dowfroth 200, 1 drop (195 g/t)

Solids	Calc Head	Cleaner Feed	Cleaner Con 1	Cleaner Con 2	Cleaner Tailing	Scavenger Tailing
Time, min	-	-	2	10	-	-
Weight, g	266.6	16.1	10.5	1.2	4.4	250.5
Analysis, %						
As	0.26	0.16	0.03	0.02	0.51	0.27
Cd	0.05	0.04	0.01	0.02	0.11	0.05
Ca	13.4	0.04	0.01	0.01	0.12	14.3
Cu	2.48	36.4	49.0	56.2	1.06	0.30
Fe	2.13	1.46	0.70	1.11	3.36	2.17
Pb	17.9	11.1	0.25	1.17	39.8	18.3
SiO ₂	7.53	2.84	0.28	0.45	9.59	7.83
Zn	1.65	3.46	1.14	4.09	8.82	1.53
Distribution, %						
As	100		0.45	0.03	3.20	96.32
Cd	100		0.80	0.18	3.68	95.33
Ca	100		0.00	0.00	0.01	99.98
Cu	100		77.75	10.19	0.70	11.36
Fe	100		1.30	0.23	2.61	95.86
Pb	100		0.06	0.03	3.68	96.24
SiO ₂	100		0.15	0.03	2.10	97.72
Zn	100		2.73	1.12	8.84	87.31

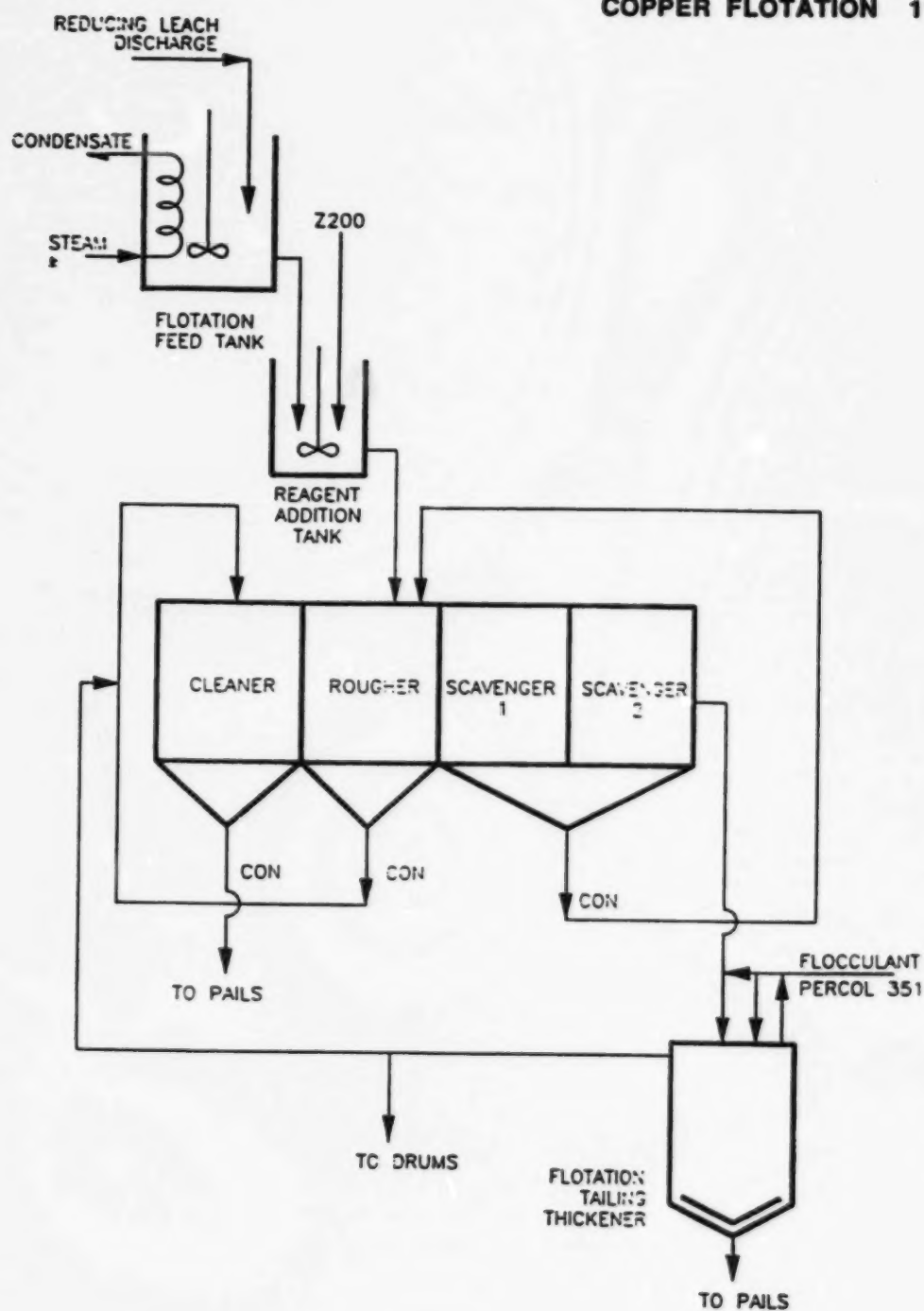


Figure 11.1 Continuous Copper Flotation Circuit

12.0 LEAD FLOTATION

Copper flotation tailing solids were treated in the lead flotation step for separation of a lead concentrate from gypsum and other gangue minerals. All lead flotation tests were carried out in a batch mode.

12.1 FEED MATERIALS

Most of the lead flotation tests were carried out with copper flotation tailing thickener underflow obtained during the continuous copper flotation test. The underflow was filtered, washed with demineralized water, and stored as a wet cake containing 42% moisture. The filter cake was repulped with demineralized water in the flotation tests.

The average analysis for this feed is provided in the table below.

Feed Solids	Analysis, %					
	Ca	Cu	Fe	Pb	SiO ₂	Zn
Copper Flotation Scavenger Tailing	12.1	0.55	0.86	25.8	6.94	1.63

Several tests were conducted with batch copper flotation tailing solids, either as a slurry directly, or as a washed cake. Analyses for these feed solids are provided with details of the individual flotation tests.

Sodium sulphide, used in Tests 1 to 4, was reagent grade (BDH). Calcium hydrosulphide was produced by bubbling H₂S through a lime slurry until all of the lime dissolved. The resulting solution contained 12.9 g/L Ca.

Hexadecyl ammonium chloride was produced by dissolving hexadecylamine (Aldrich) in ethyl ether and adding hydrogen chloride gas to precipitate the ammonium salt.

12.2 FLOTATION WITH SODIUM SULPHIDE

Lead sulphate, or anglesite, may be recovered by flotation if the surface of the lead sulphate particles is first activated by addition of a soluble sulphide, as described in Section 4. Activation is achieved at a relatively high pH, to allow for adhesion of sulphide films to the surface. Sodium hydroxide or lime was added to increase the pH of the solution in Tests 1 to 4. Sodium sulphide was then added to sulphidize the lead sulphate particles. Potassium amyl xanthate (Aero 350) was added as the collector, and lead sulphate was recovered as the flotation concentrate. Results of the first four tests are presented in Tables 12.1 to 12.4 and are summarized overleaf. The feed solids contained 26% Pb.

Test	Solids % Vol	Reagents	pH	Analysis, %				Recovery, %*	
				First Concentrate		Scavenger Tailing			
				Ca	Pb	Ca	Pb	Ca	Pb
1	1	NaOH, Na ₂ S, Aero 350	8.6	0.3	53	16	13	1	66
2	1	NaOH, Na ₂ S, Aero 350	8.9	2.7	45	20	3	13	95
3	1	NaOH, Na ₂ S, Aero 350	9.7	0.4	58	20	2	11**	94**
4	3	CaO, Na ₂ S, Aero, 350	9.8	0.4	53	23	2	7	95

* To combined rougher-scavenger concentrate

** Does not include last fraction of concentrate

Sodium hydroxide was added in the first test to bring the solution to about pH 8.5. After addition of sodium sulphide and Aero 350, good separation of lead and gypsum was achieved, with the first fraction of rougher-scavenger concentrate containing 53% Pb and only 0.3% Ca by weight. In fact, the combined rougher-scavenger concentrate contained only 1% off the calcium in the feed. Lead recovery to the concentrate, however, was only 66%.

The pH of the solution was increased to about 9.0 in the second test, and recovery of lead increased to 95%. The concentrate was of a lower grade, however, and contained 13% of the calcium in the feed.

Test 3 was carried out at pH 9.7, and 94% of the lead was recovered to a high grade concentrate, with the first cut of concentrate containing 58% by weight Pb.

Lime was used instead of sodium hydroxide in Test 4, and the results were very similar to those of the third test, with 95% recovery of lead to the concentrate. The first cut of concentrate, approximately representative of a cleaner concentrate, analyzed 53% lead.

The feed slurry in Tests 1 to 3 contained 4% solids by weight (1% by volume), which provided convenient sample sizes for work up and analysis. The slurry density was increased to 12% by weight solids in Test 4, to more closely represent commercial practice, with no reduction in lead grade or recovery.

12.3 FLOTATION WITH CALCIUM HYDROSULPHIDE

Use of lime and calcium hydrosulphide rather than sodium hydroxide and sodium sulphide in lead flotation may offer advantages in terms of lower reagent costs. Addition of sodium to the process solution may also be undesirable because of the effect of sodium in other steps in an integrated zinc plant. Several flotation tests were carried out to establish that lime and calcium hydrosulphide could be substituted for the corresponding sodium reagents in lead flotation. The results are given in Tables 12.5 to 12.10, and are summarized in the table overleaf.

Test	Solids % Vol	Reagents, g/t		pH*	Analysis, %				Recovery, %**	
					First Concentrate		Scavenger Tailing			
		Ca(HS)2	Aero 350		Ca	Pb	Ca	Pb	Ca	Pb
5	3	1300	750	9.5	3.4	46	20	5.4	17	87
6	1	400	1175	9.5	0.1	53	23	6.0	4	86
7+	0.5	-	-	9.3	0.1	58	-	-	-	-
8	1	430	1250	3.5	0.1	60	18	8.1	0.3	82
9	1	1900	2300	9.5	3.2	47	25	5.5	15	90
10	1	2100	2500	9.5	4.1	44	22	6.2	23	84

* pH adjusted with lime

** To combined rougher-scavenger concentrate

+ Cleaner flotation test

The feed material in Tests 5, 6, 8, and 9 was washed continuous copper flotation thickener underflow solids, as for Tests 1 to 4. Test 7 was a cleaner flotation of rougher concentrate slurry collected under the same conditions as Test 6. The flotation tailing from copper flotation Test 11 was the feed for lead flotation Test 10.

Lead recovery in the tests using calcium hydrosulphide as the sulphidizing agent ranged from 82% to 90%, compared with about 95% in tests with sodium sulphide. The grade of the lead concentrate was also lower in the tests with calcium hydrosulphide, although with cleaning, the concentrate contained 60% Pb and only 0.1% Ca (Test 7).

12.4 FLOTATION WITH HEXADECYL AMMONIUM CHLORIDE

Primary amines or their salts are known to promote flotation of lead sulphate, but these compounds are also known to promote flotation of other oxidic minerals such as gypsum. Hexadecyl ammonium chloride, however, has been reported to be selective for flotation of lead sulphate over gypsum, and would appear to be a good candidate for use in a process for separation of lead sulphate from gypsum and other gangue minerals. A particular advantage of such a reagent over the combination of sulphidization and xanthate addition is that the primary amines are effective at low pH, and filtration and washing of the copper flotation solids prior to lead flotation would not be necessary.

The results for tests with hexadecyl ammonium chloride as the flotation reagent are given in Tables 12.11 to 12.14 and are summarized overleaf.

Test	Solids % Vol	Reagent, g/t*	Analysis, %				Recovery, %**	
			First Concentrate		Scavenger Tailing			
			Ca	Pb	Ca	Pb	Ca	Pb
11	1	2800	2.45	59.1	4.89	5.50	92.6	96.8
12	1	1765	5.47	51.2	14.3	13.0	10.0	66.0
13	1	1720	0.23	68.7	19.2	7.24	6.6	83.7
14	1	3045	18.0	14.1	3.68	22.8	89.5	59.2

* Hexadecyl ammonium chloride

** To combined rougher-scavenger concentrate

The feed material in Tests 11 to 13 was the washed cake used in the previous lead flotation tests, repulped with 5 g/L H₂SO₄ solution. Good separation of gypsum and lead sulphate was initially achieved in Test 11, with the first three fractions of concentrate containing 55 to 62% by weight Pb and only 1 to 2% Ca. Lead recovery to these three concentrates, however, was only 59%. Two further additions of hexadecyl ammonium chloride increased the overall lead recovery to 97%, but over 90% of the gypsum also reported to the combined rougher-scavenger concentrate.

The reagent addition rate was decreased in Test 12, and only 10% of the gypsum reported to the rougher-scavenger concentrate. However, lead recovery was only 66%.

Test 13 consisted of rougher-scavenger flotation as in Test 12, followed by a cleaner flotation. The first fraction of cleaner concentrate was of a very high grade, with 69% by weight Pb and only 0.2% Ca. Overall recovery of lead to the rougher-scavenger concentrate was 84%. A higher lead recovery would likely require additional reagent, and an additional cleaning stage would then be required to produce a high grade lead concentrate.

The feed material in Test 14 was the tailing slurry from copper flotation Test 12. The flotation response of lead sulphate was very poor, with recovery of gypsum to the concentrate exceeding that of lead sulphate. The feed slurry was allowed to sit for over one month prior to conducting this flotation test. Aging of the slurry after treatment with sulphur dioxide has an impact on the performance of copper flotation, and aging may also have had an influence on this lead flotation.

12.5 CONCLUSIONS AND RECOMMENDATIONS

Lead sulphate may be recovered by flotation of the copper flotation tailing solids. With sulphidization of the solids, a lead recovery of about 95% is obtained to a flotation

concentrate containing about 60% lead by weight. Gypsum is almost wholly rejected to the flotation tailing. Effective sulphidization requires a solution pH of about 9.5, and since the copper flotation tailing solution contains a high concentration of zinc sulphate, the solids must be washed efficiently prior to adjusting the pH to avoid precipitation and loss of zinc.

The reagent hexadecyl ammonium chloride acts as a collector for lead sulphate in acidic solution, and pH adjustment of the copper flotation tailing slurry prior to lead flotation is not required if this reagent is used. Treatment of the copper flotation slurry directly with hexadecyl ammonium chloride would save a liquid-solid separation step. Flotation concentrates containing between 60 and 70% Pb were obtained with this reagent, but lead recovery was no better than 84%. A higher recovery was only possible at the expense of concentrate grade, although an additional cleaning step may allow for both high recovery and high grade. Sulphidization of lead sulphate at pH 9.5 is considered in the process selection (Section 3.0), but additional testwork with hexadecyl ammonium chloride and other collectors, such as other amines or sulphonates, should be carried out in future phases of the process development studies to determine if recovery of lead by direct flotation of the copper flotation slurry is viable.

Table 12.1 Batch Lead Flotation: Test 1

Feed Material : Continuous copper flotation tailing thickener underflow
 repulped with distilled water
 Solids Concentration : 44 g/L slurry (1% by volume)
 Slurry Volume : 2.0 L
 Solution pH : 8.65 (3.6 g NaOH added)
 Temperature : 22°C
 Reagents (Conditioning Time) : Na₂S, 740 g/t (3 min); Aero 350, 2300 g/t (3 min)
 Frothing Agent : none

Solids	Calc Head	Con 1	Con 2	Tailing
Time, min	-	3	10	-
Weight, g	87.4	16.0	11.6	59.8
Analysis, %				
As	0.14	0.30	0.36	0.06
Cd	0.05	0.10	0.10	0.03
Ca	11.1	0.31	0.49	16.1
Cu	0.55	1.18	1.10	0.28
Fe	0.96	1.29	1.30	0.80
Pb	25.8	52.8	55.1	12.9
SiO ₂	5.85	7.84	7.63	4.97
Zn	1.71	3.01	2.88	1.13
Distribution, %				
Ca	100	0.5	0.6	98.9
Fe	100	24.7	18.0	57.2
Pb	100	37.5	28.3	34.2
Zn	100	32.3	22.4	45.3

Table 12.2 Batch Lead Flotation: Test 2

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 41 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: 8.9 (4.4 g NaOH added)
Temperature	: 22°C
Reagents (Conditioning Time)	: Na ₂ S, 790 g/t (3 min); Aero 350, 2400 g/t (3 min)
Frothing Agent	: Dowfroth 200, 1 drop (250 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	2	5	10	-
Weight, g	82.4	33.6	10.5	1.5	36.8
Analysis, %					
As	0.11	0.20	0.16	0.13	<0.02
Cd	0.05	0.08	0.09	0.09	0.01
Ca	10.4	2.67	2.18	1.00	20.1
Cu	0.56	0.97	0.92	0.79	0.08
Fe	0.67	1.27	0.97	1.38	<0.005
Pb	26.4	45.1	45.6	46.7	2.92
SiO ₂	6.01	7.48	9.43	12.3	3.43
Zn	1.79	2.77	2.97	3.06	0.51
Distribution, %					
Ca	100	10.5	2.7	0.2	86.6
Fe	100	77.4	18.5	3.8	<0.3
Pb	100	69.8	22.0	3.2	5.0
Zn	100	63.0	21.1	3.1	12.7

Table 12.3 Batch Lead Flotation: Test 3

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 41 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH	: 9.7 (7.1 g NaOH added)
Temperature	: 22°C
Reagents (Conditioning Time)	: Na ₂ S, 800 g/t (3 min); Aero 350, 2500 g/t (3 min)
Frothing Agent	: Dowfroth 200, 1 drop (260 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	5	10	-
Weight, g	81.4	17.9	11.3	12.3	11.1	28.8
Analysis, %						
As	0.12	0.28	0.26	0.07	0.06	<0.02
Cd	0.05	0.10	0.10	0.07	0.02	0.01
Ca	10.9	0.38	1.05	6.68	18.6	20.2
Cu	0.57	1.07	1.11	0.79	0.17	0.10
Fe	0.47	0.88	1.02	0.78	0.10	<0.005
Pb	27.0	57.8	54.1	34.9	6.74	1.72
SiO ₂	6.39	8.94	9.75	8.72	4.35	3.28
Zn	1.79	2.99	3.12	2.48	0.85	0.60
Distribution, %						
Ca	100	0.8	1.3	9.2	23.2	65.4
Fe	100	41.3	30.2	25.2	2.9	<0.4
Pb	100	47.0	27.8	19.5	3.4	2.2
Zn	100	36.7	24.2	20.9	6.5	11.8

Table 12.4 Batch Lead Flotation: Test 4

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 122 g/L slurry (3% by volume)
Slurry Volume	: 2.0 L
Solution pH	: 9.8 (79.3 g 200 g/L CaO slurry added)
Temperature	: 22°C
Reagents (Conditioning Time)	: Na ₂ S, 660 g/t (3 min); Aero 350, 800 g/t (3 min)
Frothing Agent	: Dowfroth 200, 1 drop (85 g/t)

Solids	Calc Head*	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	5	10	-
Weight, g	244.8	21.6	24.8	39.5	21.7	137.2
Analysis, %						
As	0.42	0.98	1.01	0.86	0.51	0.09
Cd	0.09	0.10	0.10	0.10	0.08	0.08
Ca	13.7	0.40	0.98	1.49	6.89	22.7
Cu	0.50	1.12	1.13	1.08	0.77	0.07
Fe	0.86	1.41	1.49	1.50	1.34	0.40
Pb	22.5	52.9	52.1	50.1	37.5	2.10
SiO ₂	6.37	10.3	10.9	10.8	9.28	3.20
S(SO ₄)	12.9	2.64	2.97	3.42	7.58	19.9
S	13.3	3.91	4.19	4.60	8.06	19.8
Zn	1.67	3.17	3.27	3.30	2.70	0.51
Distribution, %						
Ca	100	0.3	0.7	1.8	4.5	92.8
Fe	100	14.5	17.5	28.1	13.8	26.1
Pb	100	20.7	23.4	35.9	14.8	5.2
Zn	100	16.8	19.8	31.9	14.3	17.1

* Includes lime

Table 12.5 Batch Lead Flotation: Test 5

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 133 g/L slurry (3% by volume)
Slurry Volume	: 2.0 L
Solution pH	: 9.5 (73 g of 200 g/L CaO added)
Temperature	: 22°C
Reagents (Conditioning Time)	: Ca(HS) ₂ , 1300 g/t (3 min); Aero 350, 750 g/t (3 min)
Frothing Agent	: Dowfroth 200, 1 drop (80 g/t)

Solids	Calc Head*	Con 1	Con 2	Con 3	Con 4	Con 5	Tailing
Time, min	-	1	2	5	10	18	-
Weight, g	265.1	19.2	24.2	45.1	24.4	8.5	143.7
Analysis, %							
As	0.12	0.26	0.25	0.23	0.29	0.11	<0.02
Cd	0.05	0.10	0.09	0.08	0.10	0.08	0.01
Ca	13.4	3.35	5.48	6.23	2.87	6.43	20.5
Cu	0.48	1.07	0.96	0.91	1.05	0.95	0.06
Fe	0.83	1.56	1.36	1.26	1.40	1.44	0.38
Pb	22.5	45.8	40.8	39.8	49.5	36.8	5.39
SiO ₂	6.41	11.0	10.4	9.66	10.6	9.92	3.19
Zn	1.46	2.83	2.60	2.52	2.94	3.21	0.40
Distribution, %							
Ca	100	1.8	3.7	7.9	2.0	1.5	83.0
Fe	100	13.6	14.9	25.7	15.5	5.5	24.7
Pb	100	14.8	16.6	30.1	20.3	5.2	13.0
Zn	100	14.0	16.2	29.3	18.5	7.0	14.8

* includes lime

Table 12.6 Batch Lead Flotation: Test 6

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 43 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH	: 9.5 (29.8 g of 200 g/L CaO added)
Temperature	: 22°C
Reagents (Conditioning Time)	: Ca(HS) ₂ , 400 g/t (3 min); Aero 350, 1175 g/t (3 min)
Frothing Agent	: Dowfroth 200, 1 drop (250 g/t)

Solids	Calc Head*	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	5	10	-
Weight, g	85.2	5.7	6.4	14.0	9.6	49.5
Analysis, %						
As	0.15	0.49	0.44	0.34	0.12	<0.02
Cd	0.05	0.13	0.12	0.11	0.08	0.01
Ca	13.7	0.07	0.07	0.06	4.73	22.6
Cu	0.54	1.41	1.36	1.15	0.76	0.12
Fe	0.95	1.82	1.75	1.58	1.37	0.49
Pb	25.8	53.2	54.8	56.8	46.5	6.03
SiO ₂	7.27	13.1	12.9	11.4	10.4	4.09
Zn	1.72	3.94	3.69	3.26	2.42	0.64
Distribution, %						
Ca	100	<0.1	<0.1	0.1	3.9	96.0
Fe	100	12.8	13.8	27.3	16.2	29.9
Pb	100	13.8	16.0	36.2	20.4	13.6
Zn	100	15.3	16.1	31.1	15.8	21.6

* Includes lime

Table 12.7 Batch Lead Flotation: Test 7 (Cleaner)

Feed Material : Rougher concentrate obtained as for Test 6 (0 to 2 min),
 repulped with distilled water
 Solids Concentration : 22 g/L slurry (0.5% by volume)
 Slurry Volume : 2.0 L
 Solution pH : 9.3
 Temperature : 22°C
 Reagents (Conditioning Time) : none
 Frothing Agent : Dowfroth 200, 1 drop (480 g/t)

Solids	Calc Head	Cleaner Con 1	Cleaner Con 2	Cleaner Con 3	Cleaner Tailing
Time, min	-	1	2	5	-
Weight, g	44.0	13.3	10.4	16.2	4.1
Analysis, %					
As	0.32	0.37	0.35	0.29	0.16
Cd	0.11	0.11	0.11	0.10	0.10
Ca	0.18	0.10	0.03	0.02	1.42
Cu	1.16	1.17	1.11	1.02	1.78
Fe	1.59	1.47	1.47	1.51	2.63
Pb	56.3	58.4	59.0	58.5	33.5
SiO ₂	11.9	10.6	10.6	11.0	22.7
Zn	3.26	3.19	3.12	3.07	4.64
Distribution, %					
Ca	100	17.1	4.0	4.2	74.8
Fe	100	27.9	21.8	34.9	15.4
Pb	100	31.4	24.8	38.3	5.5
Zn	100	29.5	22.6	34.6	13.2

n.a. = not analyzed

Table 12.8 Batch Lead Flotation: Test 8

Feed Material : Continuous copper flotation tailing thickener underflow
 repulped with distilled water
 Solids Concentration : 40 g/L slurry (1% by volume)
 Slurry Volume : 2.0 L
 Solution pH : 3.5 (no neutralizing agent added)
 Temperature : 22°C
 Reagents (Conditioning Time) : Ca(HS)₂, 430 g/t (3 min); Aero 350, 1250 g/t (3 min)
 Frothing Agent : Dowfroth 200, 1 drop (260 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	2	5	10	-
Weight, g	79.8	16.6	9.0	4.9	49.3
Analysis, %					
As	0.07	0.12	0.10	0.11	0.04
Cd	0.06	0.10	0.09	0.09	0.03
Ca	11.1	0.09	0.08	0.11	17.9
Cu	0.56	1.16	0.82	0.78	0.29
Fe	0.84	0.51	0.50	0.86	1.01
Pb	27.5	60.2	59.0	53.7	8.07
SiO ₂	7.39	1.37	2.55	6.39	10.4
Zn	1.49	1.67	1.44	1.91	1.39
Distribution, %					
Ca	100	0.2	0.1	0.1	99.7
Fe	100	12.6	6.7	6.3	74.3
Pb	100	45.6	24.2	12.0	18.2
Zn	100	23.4	10.9	7.9	57.8

Table 12.9 Batch Lead Flotation: Test 9

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 45 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH	: 9.5 (63 g of 100 g/L CaO slurry added)
Temperature	: 22°C
Reagents (Conditioning Time)	: Ca(HS) ₂ , 1900 g/t (3 min); Aero 350, 2300 g/t (3 min)
Frothing Agent	: Dowfroth 200, 1 drop (230 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Tailing
Time, min	-	1	5	11	-
Weight, g	89.7	28.0	12.1	5.3	44.3
Analysis, %					
As	0.36	0.21	0.20	0.24	0.51
Cd	0.06	0.09	0.09	0.05	0.03
Ca	14.6	3.21	2.22	15.3	25.1
Cu	0.64	1.18	1.02	0.64	0.20
Fe	1.10	1.42	1.44	1.13	0.80
Pb	25.8	47.4	51.6	23.1	5.49
SiO ₂	8.06	11.8	11.0	8.29	4.86
Zn	1.71	2.92	2.74	1.69	0.67
Distribution, %					
As	100	18.3	7.5	4.0	70.2
Cd	100	48.4	20.9	5.1	25.5
Ca	100	6.9	2.1	6.2	84.9
Cu	100	57.3	21.4	5.9	15.4
Fe	100	40.3	17.7	6.1	35.9
Pb	100	57.3	26.9	5.3	10.5
SiO ₂	100	45.7	18.4	6.1	29.8
Zn	100	53.2	21.6	5.8	19.3

Table 12.10 Batch Lead Flotation: Test 10

Feed Material : Copper flotation tailing solids from Batch Test 11, repulped with distilled water
 Solids Concentration : 40 g/L slurry (1% by volume)
 Slurry Volume : 2.0 L
 Solution pH : 9.5 (40 g of 100 g/L CaO slurry added)
 Temperature : 22°C
 Reagents (Conditioning Time) : Ca(HS)₂, 2100 g/t (3 min); Aero 350, 2500 g/t (3 min)
 Frothing Agent : Dowfroth 200, 1 drop (260 g/t)

Solids	Calc Head ^a	Con 1	Con 2	Con 3	Tailing
Time, min	-	2	5	11	-
Weight, g	81.0	21.6	7.4	9.0	43.0
Analysis, %					
As	0.48	1.12	0.98	0.26	0.13
Cd	0.06	0.12	0.11	0.04	0.02
Ca	15.1	4.10	3.89	18.4	21.8
Cu	0.35	0.72	0.65	0.22	0.13
Fe	2.37	3.94	3.70	1.43	1.54
Pb	20.8	44.3	43.8	15.6	6.19
SiO ₂	5.99	6.13	7.01	4.54	6.04
Zn	1.70	3.13	3.04	1.10	0.88
Distribution, %					
As	100	61.7	18.5	5.9	13.9
Cd	100	54.7	17.0	7.1	21.2
Ca	100	7.3	2.4	13.6	76.8
Cu	100	55.5	17.0	6.9	20.5
Fe	100	44.4	14.3	6.7	34.6
Pb	100	56.7	19.2	8.3	15.8
SiO ₂	100	27.3	10.7	8.4	53.6
Zn	100	49.1	16.3	7.2	27.4

Table 12.11 Batch Lead Flotation: Test 11

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 43 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 22°C
Reagents (Conditioning Time)	: Hexadecyl ammonium chloride, 2800 g/t (3 min), added in three equal portions at 0, 5, and 10 min
Frothing Agent	: Dowfroth 200, 2 drops (520 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Con 5	Tailing
Time, min	-	1	2	5	10	15	-
Weight, g	86.6	12.2	5.7	6.5	41.5	6.8	13.9
Analysis, %							
As	0.12	0.05	0.06	0.15	0.07	0.07	0.39
Cd	0.06	0.09	0.09	0.10	0.04	0.03	0.07
Ca	10.6	2.45	1.38	1.50	16.7	16.4	4.89
Cu	0.56	0.42	0.69	1.12	0.15	0.48	1.60
Fe	0.91	0.31	0.41	0.84	0.22	0.62	3.90
Pb	27.9	59.1	61.9	55.2	20.1	10.9	5.50
SiO ₂	7.12	1.39	1.39	4.19	1.37	4.99	34.1
S(SO ₄)	11.5	9.53	9.70	9.45	14.6	14.5	4.05
Zn	1.51	0.89	1.36	2.88	0.46	1.17	4.78
Distribution, %							
As	100	5.4	3.2	8.9	27.7	4.2	50.5
Cd	100	21.2	10.7	12.4	31.4	3.8	20.5
Ca	100	3.2	0.9	1.1	75.3	12.1	7.4
Cu	100	10.5	8.2	15.1	13.3	6.7	46.2
Fe	100	4.8	2.9	6.9	11.5	5.3	68.5
Pb	100	29.8	14.6	14.8	34.5	3.1	3.2
SiO ₂	100	2.7	1.3	4.4	9.2	5.5	76.8
S(SO ₄)	100	11.7	5.6	6.2	61.0	9.9	5.7
Zn	100	8.3	5.9	14.3	14.5	6.1	50.8

Table 12.12 Batch Lead Flotation: Test 12

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: 43 g/L slurry (1% by volume)
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 22°C
Reagents (Conditioning Time)	: Hexadecyl ammonium chloride, 1765 g/t (3 min), added in one portion
Frothing Agent	: Dowfroth 200, 1 drop (250 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	5	10	-
Weight, g	85	12.3	5.8	6.1	2.9	57.9
Analysis, %						
As	0.23	0.06	0.08	0.28	0.13	0.28
Cd	0.06	0.08	0.09	0.10	0.09	0.04
Ca	10.8	5.47	3.14	0.13	2.14	14.3
Cu	0.56	0.43	0.61	1.31	0.78	0.49
Fe	0.95	0.32	0.40	0.86	0.57	1.17
Pb	26.1	51.2	56.1	56.4	56.6	13.0
SiO ₂	7.77	1.22	1.52	6.26	2.72	10.2
S(SO ₄)	12.8	12.3	11.3	9.11	10.6	13.6
Zn	1.53	0.95	1.40	1.97	1.83	1.6
Distribution, %						
As	100	3.8	2.4	8.8	1.9	83.1
Cd	100	19.8	10.3	12.3	5.5	52.1
Ca	100	7.3	2.0	0.1	0.7	90.0
Cu	100	11.1	7.4	16.8	4.7	60.0
Fe	100	4.8	2.9	6.5	2.0	83.8
Pb	100	28.4	14.7	15.5	7.4	34.0
SiO ₂	100	2.3	1.3	5.8	1.2	89.4
S(SO ₄)	100	13.9	6.0	5.1	2.8	72.2
Zn	100	9.0	6.3	9.3	4.1	71.4

Table 12.13 Batch Lead Flotation: Test 13

Feed Material	: Continuous copper flotation tailing thickener underflow repulped with distilled water
Solids Concentration	: rougher-scavenger 87 g/L; cleaner 35 g/L
Slurry Volume	: 2.0 L
Solution pH (g/L H ₂ SO ₄)	: (5 g/L)
Temperature	: 22°C
Reagents (Conditioning Time)	: Hexadecyl ammonium chloride, 1720 g/t (3 min), added in one portion in rougher-scavenger test
Frothing Agent	: Dowfroth 200, 1 drop (250 g/t) to both rougher and cleaner

Solids	Calc Head	Cleaner Con 1	Cleaner Con 2	Cleaner Con 3	Cleaner Tailing	Rough-Scav Tailing
Time, min	-	1	2	5	-	-
Weight, g	174.2	29.2	15.3	10.7	14.0	105.0
Analysis, %						
As	0.14	0.09	0.02	0.08	0.30	0.16
Cd	0.05	0.10	0.08	0.09	0.06	0.03
Ca	12.4	0.23	0.17	0.75	8.86	19.2
Cu	0.53	0.32	0.58	0.77	1.09	0.48
Fe	0.91	0.28	0.31	0.49	0.90	1.22
Pb	26.7	68.7	57.3	62.7	24.7	7.24
SiO ₂	7.68	0.94	0.88	1.84	7.99	11.1
S(SO ₄)	13.7	10.3	9.9	10.1	11.7	15.8
Zn	1.52	0.84	1.32	1.82	1.82	1.67
Distribution, %						
As	100	10.6	1.2	3.5	16.9	67.8
Cd	100	30.0	13.4	10.6	8.1	37.8
Cu	100	0.3	0.1	0.4	5.7	93.4
Ca	100	10.0	9.6	8.9	16.5	54.9
Fe	100	5.2	3.0	3.3	7.9	80.7
Pb	100	43.1	18.8	14.4	7.4	16.3
SiO ₂	100	2.1	1.0	1.5	8.4	87.1
S(SO ₄)	100	12.6	6.4	4.5	6.9	69.6
Zn	100	9.3	7.6	7.3	9.6	66.1

Table 12.14 Batch Lead Flotation: Test 14

Feed Material : Flotation tailing slurry from copper flotation Batch Test 12
 Solids Concentration : 35 g/L slurry (1% by volume)
 Slurry Volume : 2.0 L
 Solution pH (g/L H₂SO₄) : (5 g/L)
 Temperature : 22°C
 Reagents (Conditioning Time) : Hexadecyl ammonium chloride, 3045 g/t (3 min), added in one portion
 Frothing Agent : Dowfroth 200, 1 drop (320 g/t)

Solids	Calc Head	Con 1	Con 2	Con 3	Con 4	Tailing
Time, min	-	1	2	5	9	-
Weight, g	65.7	19.7	6.7	12.4	4.3	22.6
Analysis, %						
As	0.33	0.08	0.11	0.20	0.46	0.65
Cd	0.05	0.03	0.03	0.04	0.05	0.08
Ca	12.1	18.0	18.5	14.9	11.2	3.68
Cu	0.32	0.08	0.09	0.16	0.33	0.67
Fe	2.54	0.36	0.40	0.84	1.87	6.13
Pb	19.2	14.1	16.6	20.2	25.3	22.8
SiO ₂	9.60	1.07	0.88	2.23	3.30	24.9
S(SO ₄)	12.0	15.5	16.7	14.9	12.7	5.96
Zn	1.73	0.42	0.48	0.81	1.54	3.78
Distribution, %						
As	100	7.3	3.4	11.6	9.2	68.4
Cd	100	15.4	6.5	14.8	7.3	56.0
Cu	100	44.6	15.6	23.3	6.1	10.5
Ca	100	7.5	3.0	9.6	6.8	73.1
Fe	100	4.3	1.6	6.2	4.8	83.1
Pb	100	22.0	8.8	19.8	8.6	40.8
SiO ₂	100	3.3	0.9	4.4	2.3	89.1
S(SO ₄)	100	38.6	14.1	23.3	6.9	17.0
Zn	100	7.3	2.8	8.9	5.8	75.1

13.0 IRON REMOVAL

Zinc sulphate solution from the reducing leach was separated from the solids in the continuous copper flotation step as copper flotation tailing thickener overflow. This overflow solution was treated with zinc hydroxide sludge and lime in two stages in batch iron removal tests. The solution in the first stage was maintained at about pH 3.5. After filtration, the first stage solution was treated at pH 5.0 in the second stage.

The objectives of the iron removal step were:

to neutralize acid and precipitate iron from the zinc sulphate solution and produce a solution suitable as feed for conventional purification and electrowinning processes;

to produce an iron removal precipitate, the first stage solids, amenable to separation from the zinc sulphate solution and to disposal by conventional techniques; and

to minimize zinc loss to the precipitated solids.

13.1 FEED MATERIALS

The copper flotation tailing thickener overflow solution contained 82 g/L Zn and 38 g/L Fe^{2+} . More detailed analyses are provided in Tables 1 and 2.

Zinc hydroxide sludge, obtained from HBM&S, was added as a slurry containing 25% by weight solids, or as a filter cake containing 43% solids. The solids analyses were given in Table 5.4, and are summarized below.

Feed Solids	Analysis, %				
	Ca	Cu	Fe	S	Zn
Zinc Hydroxide Sludge	11.8	0.20	0.10	12.8	26.7

Lime was added as a 200 g/L slurry for neutralization in the second stage of iron removal.

13.2 RESULTS

The results for the two iron removal tests are given in Tables 13.1 and 13.2. Both tests were conducted at 85°C and at atmospheric pressure. Oxygen was sparged into the vessel at a rate of 0.5 L/min in the first stage of iron removal, and 0.2 L/min in the second stage. Dual radial impellers were employed to provide good gas dispersion.

The iron removal process is described in some detail in Section 3 and is similar to that proposed for the HBM&S zinc pressure leach plant.

Acid is neutralized and the bulk of the iron is precipitated in the first stage of iron removal, at a solution pH of about 3.5. This pH is chosen to provide a rapid iron oxidation rate, good liquid-solid separation characteristics, and minimal precipitation of zinc hydroxide. Iron in the feed solution is ideally in the ferrous state, as ferric iron in the feed leads to slimy precipitates with poor liquid-solid separation characteristics. The first stage iron removal solids are separated from the solution and impounded. The solution proceeds to the second stage, which is operated at pH 5 to allow for precipitation of most of the remaining iron in the solution. Since some zinc is also precipitated at this pH, the second stage solids are recycled to the first stage to redissolve and recover the zinc.

13.2.1 Solution Quality

The feed solution in the iron removal tests contained about 40 g/L Fe, compared with about 2 g/L Fe in the zinc pressure leach testwork carried out for HBM&S. Nevertheless, most of this iron was precipitated within three or four hours at pH 3.5 in the two batch tests. The second stage solution contained less than 1 mg/L Fe after one or two hours at pH 5.0. The results are plotted in Figure 13.1 and are summarized below.

Test	Solution Analysis, g/L								
	Feed			First Stage			Second Stage		
	As	Fe	Zn	As	Fe	Zn	As	Fe	Zn
1	2.2	38.2	81.5	<0.02	0.1	94.2	0.0009	<0.0005	85.1
2	2.2	38.2	81.5	<0.02	0.1	104	0.0003	<0.0005	114

Zinc hydroxide sludge was used for neutralizing the first stage solution in both tests. A 25% solids slurry was used in the first test, while a 43% solids filter cake was used in the second. The zinc concentration in the product solution was correspondingly higher in the second test.

Arsenic and other impurity elements are precipitated with iron in the iron removal process. The deportment of these elements is described in more detail in Section 4.

13.2.2 Reagent Consumption

Consumptions of zinc hydroxide sludge and lime were monitored in the second test. The consumptions were 367 g of zinc hydroxide sludge solids in the first stage and 6 g of lime in the second stage. The stoichiometric requirement for 1.5 L of first stage feed solution containing 7.5 g/L H_2SO_4 and 38.2 g/L Fe^{2+} is 64 g CaO. The lime equivalent of the zinc hydroxide sludge, determined by acid titration, was 185 g CaO/kg, which is in good agreement with the iron removal test.

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Sludge consumption was 552 kg per tonne of oxide residue (dry basis) according to the material balance outlined in Section 4. This would correspond to a lime consumption of about 100 kg/t.

13.2.3 Solids Composition

Analyses of the first and second stage solids for the batch tests are summarized below.

Test	Solids Analysis, %							
	First Stage					Second Stage		
	As	Ca	Fe	S(SO ₄)	Zn	Ca	Fe	Zn
1	1.4	14.2	20.9	12.5	0.8	n.a.	n.a.	n.a.
2	1.3	15.3	22.5	13.5	1.1	0.03	9.9	48.4

n.a. = not analyzed

The first stage solids contained about 20% iron and 1% zinc. Zinc loss to these solids represented about 2% of the zinc in the feed solution to iron removal, or about 5% of the zinc in the oxide residue feed, as described in Section 4. The solids also contained about 1.4% arsenic and 13% sulphur as sulphate. About 90% of the sulphate could be accounted for as gypsum. Most of the remainder, about 1% by weight of the first stage solids, is likely present as basic iron or zinc sulphate.

If lime or limestone were to be used for neutralization instead of zinc hydroxide sludge, it is expected that the first stage solids would contain about 17% Fe, as observed in the Phase Ia oxide residue study. Since the ratio of zinc to iron in the precipitate would be expected to be constant, these solids would then contain about 0.75% Zn.

The second stage solids contained nearly 50% by weight zinc. This represented about 2% of the zinc in the oxide residue feed, which can be recovered by recycle of the second stage solids to the first stage of the iron removal process.

13.3 CONCLUSIONS AND RECOMMENDATIONS

The two stage iron removal process is effective in precipitating iron from zinc sulphate solution. With proper control of pH, the first stage (discard) iron solids contain less than 1% Zn by weight, representing about 2% of the zinc in the total feed solids and about 5% of the zinc in the oxide residue feed. The second stage solution contains greater than 100 g/L Zn and less than 5 mg/L Fe, and is suitable as feed to conventional zinc purification and electrowinning steps.

Good oxygen dispersion is essential for rapid iron precipitation. Under oxygen sparging conditions, 40 g/L Fe may be precipitated from solution within three to four hours at 85°C and at atmospheric pressure. Maintaining the solution at pH 3.5 in the first stage of iron removal allows for minimum precipitation of zinc and good liquid-solid separation characteristics. A solution pH of 5.0 in the second stage ensures nearly complete removal of iron, but precipitation of zinc is significant, and the second stage solids should be recycled to the first stage for optimum zinc recovery.

Table 13.1 Iron Removal Batch Test 1

Feed Solution	:	Continuous copper flotation tailing thickener overflow
Volume	:	1.5 L
Temperature	:	85°C
Oxidant	:	Oxygen sparge, first stage 0.5 L/min; second stage 0.2 L/min
Equipment	:	Cylindrical glass vessel, 10.5 cm diameter x 29 cm in height, equipped with cover, baffles, heating tape and sparge tube with Inlet below bottom impeller
Agitation	:	Dual radial impellers, 7.6 cm diameter, 1300 rev/min
Neutralizing Agents	:	First stage, zinc hydroxide sludge slurry, 25% solids Second stage, lime slurry, 200 g/L CaO

Analysis	Retention Time, h						
	First Stage						Second Stage
	Head	0.5	1.0	2.0	3.0	4.0	1.0
Solution, g/L							
As	2.19	0.03	<0.02	<0.02	<0.02	<0.02	0.0009
Cd	0.14	0.15	0.14	0.16	0.16	0.17	0.15
Ca	0.28	0.50	0.50	0.49	0.51	0.51	0.51
Cu	0.24	<0.01	0.02	<0.01	0.06	0.09	0.05
Fe ²⁺	n.a.	15.1	8.3	6.9	3.5	0.1	n.a.
Fe	38.2	14.3	7.90	6.64	3.27	0.08	0.0004
H ₂ SO ₄ (pH)	7.5	(4.3)	(3.6)	(3.4)	(3.4)	(4.2)	(5.5)
Zn	81.5	90.9	81.8	91.4	91.9	94.2	85.1
Solids, %							
As	n.a.	n.a.	n.a.	n.a.	1.36	n.a.	n.a.
Cd	n.a.	n.a.	n.a.	n.a.	0.01	n.a.	n.a.
Ca	n.a.	n.a.	n.a.	n.a.	14.2	n.a.	n.a.
Cu	n.a.	n.a.	n.a.	n.a.	0.30	n.a.	n.a.
Fe	n.a.	n.a.	n.a.	n.a.	20.9	n.a.	n.a.
SiO ₂	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
S(SO ₄)	n.a.	n.a.	n.a.	n.a.	12.5	n.a.	n.a.
Zn	n.a.	n.a.	n.a.	n.a.	0.78	n.a.	n.a.

n.a. = not analyzed

Table 13.2 Iron Removal Batch Test 2

Feed Solution	:	Continuous copper flotation tailing thickener overflow
Volume	:	1.5 L
Temperature	:	85°C
Oxidant	:	Oxygen sparge, first stage 0.5 L/min; second stage 0.2 L/min
Equipment	:	Cylindrical glass vessel, 10.5 cm diameter x 29 cm in height, equipped with cover, baffles, heating tape and sparge tube with inlet below bottom impeller
Agitation	:	Dual radial impellers, 7.6 cm diameter, 1300 rev/min
Neutralizing Agents	:	First stage, zinc hydroxide sludge cake, 43% solids Second stage, lime slurry, 200 g/L Cao

Analysis	Retention Time, h							
	First Stage						Second Stage	
	Head	0.5	1.0	2.0	3.0	4.0	1.0	2.0
Solution, g/L								
As	2.19	0.04	<0.02	<0.02	<0.02	<0.02	0.0004	0.0003
Cd	0.14	0.14	0.16	0.18	0.18	0.18	0.17	0.18
Ca	0.28	0.44	0.55	0.56	0.39	0.45	0.72	0.81
Cu	0.24	<0.01	<0.01	<0.01	0.09	0.19	0.14	0.13
Fe ²⁺	n.a.	23.1	13.1	3.3	0.2	0.2	n.a.	n.a.
Fe	38.2	21.5	12.6	3.14	0.10	0.10	0.0042	<0.0005
H ₂ SO ₄ (pH)	7.5	(3.7)	(3.5)	(3.7)	(3.8)	(3.6)	(5.2)	(5.1)
Zn	81.5	101	105	111	100	104	110	114
Solids, %								
As	n.a.	n.a.	n.a.	n.a.	n.a.	1.32	n.a.	n.a.
Cd	n.a.	n.a.	n.a.	n.a.	n.a.	0.02	n.a.	0.11
Ca	n.a.	n.a.	n.a.	n.a.	n.a.	15.3	n.a.	0.03
Cu	n.a.	n.a.	n.a.	n.a.	n.a.	0.26	n.a.	0.26
Fe	n.a.	n.a.	n.a.	n.a.	n.a.	22.5	n.a.	9.90
SiO ₂	n.a.	n.a.	n.a.	n.a.	n.a.	0.60	n.a.	0.36
S(SO ₄)	n.a.	n.a.	n.a.	n.a.	n.a.	13.5	n.a.	n.a.
Zn	n.a.	n.a.	n.a.	n.a.	n.a.	1.12	n.a.	48.4

Reagent Consumption: 367 g zinc hydroxide sludge solids, first stage
6 g CaO, second stage

n.a. = not analyzed

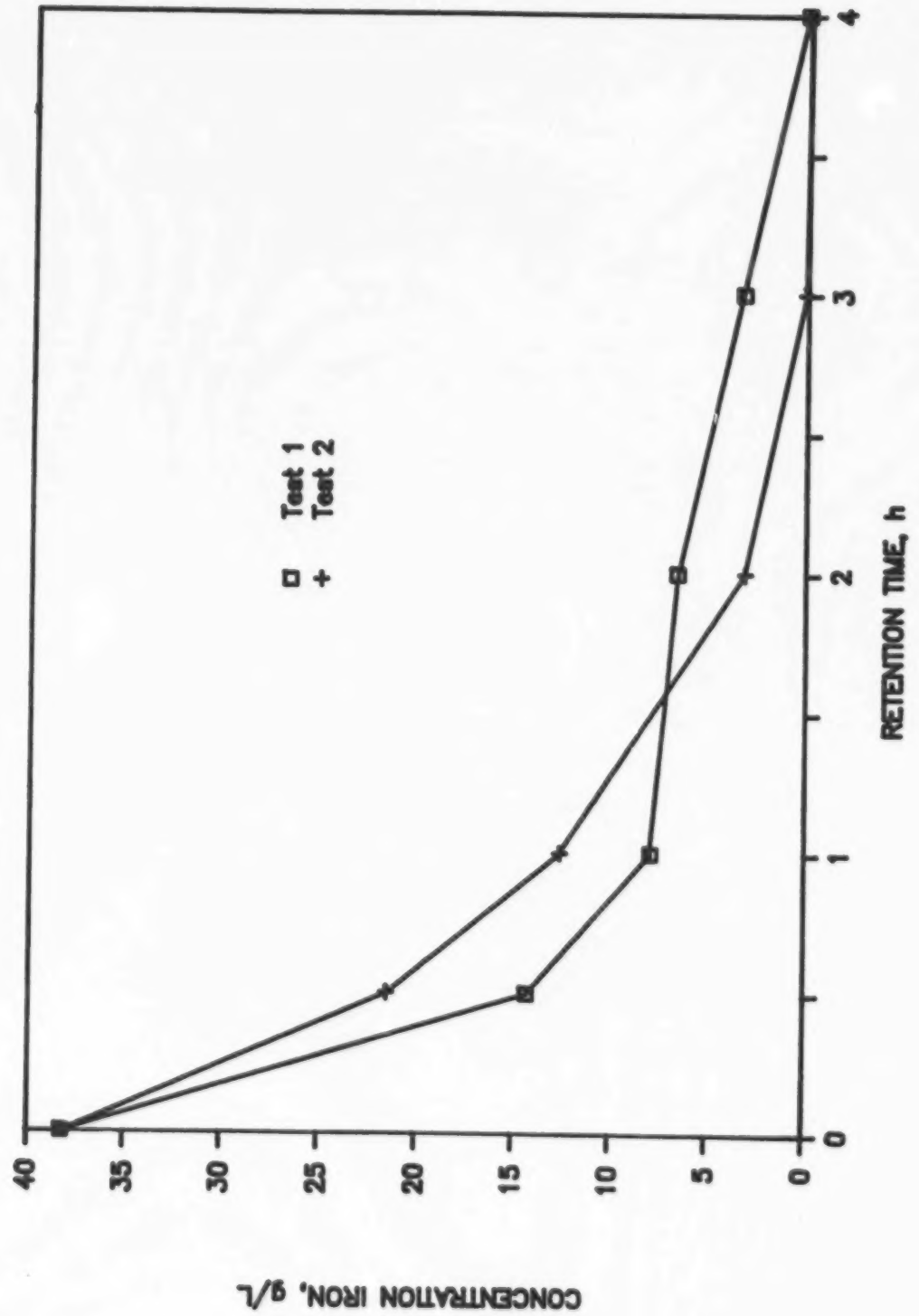


Figure 13.1 Iron In First Stage Solution